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AIR DRYING, HIGH TEMPERATURE RESISTANT, SILICONE PROTECTIVE COATINGS

R. L. STOUT

TECHNICAL REPORT AFML-TR-67-433

APRIL 1968

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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The distribution of this document is limited because of the advancement of the state-of-the-art in the high temperature protective coatings area.

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FOREWORD

This report was prepared by the Elastomers and Coatings Branch, Nonmetallic Materials Division, Air Force Materials Laboratory. The work was conducted under Project No. 7340 "Nonmetallic and Composite Materials", Task 734007 "Coatings for Energy Utilization Control and Protective Functions", with Mr. R. L. Stout acting as project engineer.

This report covers work from July 1962 to October 1964 including the initial research, two years of Florida climatic exposure and the final laboratory evaluation. This manuscript was released by the author December 1967 for publication as an AFML Technical Report.

None of the materials used in this project were developed or intended by the manufacturer for the conditions to which they were subjected. Any failure or poor performance of a material is, therefore, not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

The author wishes to acknowledge the assistance of Mr. Henry Maas, of the University of Dayton Research Institute.

This technical report has been reviewed and is approved.

Warren P. Johnson

WARREN P. JOHNSON, Acting Chief
Elastomers and Coatings Branch
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ABSTRACT

Improved high temperature protective coatings primarily for use on high speed Mach 3 aircraft and missiles have been developed which are capable of withstanding the extreme environments and aerodynamic heating. By selectively incorporating aminosilanes as catalysts for curing unmodified polymethylphenyl silicone resins, air dry (ambient temperature), stable coatings with retained reflectances exceeding eighty (80) percent after elevated temperature exposures were developed. Analysis of two years Florida weathering data indicates that these coatings when properly applied to titanium, stainless steel and aluminum alloys have excellent adhesion, corrosion resistance, and are extremely resistant to solar discoloration thus making them excellent candidates for high speed aircraft and missiles. A variety of air dry silicone primer systems were also developed, evaluated for thermal stability and corrosion resistance, and optimized for the best topcoats formulated. Based on the laboratory and Florida weathering results, a silicone-base coating system which will dry under ambient temperature conditions (75 ± 2°F), and serviceable for use up to 700°F for short periods and 600°F for prolonged periods has been developed.

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SECTION I

INTRODUCTION

When aircraft first broke the sound barrier they created a series of materials problems heretofore unencountered. One such problem is that of aerodynamic heating. This heating results in external skin leading edge stagnation temperatures exceeding 200°F for Mach 1 during straight and level flight progressing upwards to 350°F for Mach 2 and exceeding 600°F for Mach 3 (exact temperature depending primarily upon altitude and surface emittance). With this increase in speed and surface temperature came the necessity of replacing the excellent and commonly used aluminum alloys and substituting the higher strength stainless steels and titanium alloys. The use of these alloys created still further problems since the heat they absorb must be dissipated both during flight and on the ground.

One practical method of minimizing the heat build-up is to provide a protective coating which will both reflect and emit as much energy as possible (depending upon the desired optical properties of the coating) and also provide corrosion resistance.

With the advent of nuclear weapons, research was conducted to develop a coating which would, when exposed to a nuclear blast, reflect as much energy from the coated surfaces as possible. As a result of this research, it was found that the most effective coatings were highly pigmented untinted "whites". Based on this information the only practical method of minimizing aerodynamic heat build up and thermonuclear flash is to provide a white protective coating capable of being applied to large structures. The exact amount of energy emitted depends upon the thermal and optical properties of the coating as well as the geometry of the aircraft.

With the use of the highly corrosion resistant steels, the necessity of using a primer for protection against corrosion appears of secondary importance and is essential only if the top coat so demands. However, more subtle types of corrosion (e.g., stress corrosion) are known to occur particularly in high strength metals even though the "rust" type corrosion is not a major problem.

In order to fulfill the 600°F high temperature requirements as set forth by Mach 3 and greater aircraft and missiles a coating should have the additional following characteristics:

1. Maintain an average spectral reflectance exceeding 80% after 100 hours exposure to elevated temperature

and a spectral normal emittance exceeding 0.80.

2. Ease of application to the substrate.
3. Capable of air drying.
4. Good adhesion and flexibility to both primed and unprimed substrates of titanium and stainless steel.
5. Good gloss and color retention before and after temperature exposure.
6. Good weathering characteristics after one year of Florida exposure.

SECTION II

SUMMARY AND CONCLUSIONS

SUMMARY

A white, highly reflective, emissive, high gloss, high temperature silicone primer and topcoat combination has been developed which can meet the requirements for use on high speed (Mach 3) aircraft. Formulation No. AF-66 consists of an unmodified silicone resin (SR-123) catalyzed with an amino-functional silane A-1100 at 10 percent based on total resin solids. This coating, when pigmented with Ti-Pure R-900 titanium dioxide at a pigment/binder ratio of 25/100, will exceed the temperature requirements needed for aircraft speeds through Mach 3. The thermal resistance of this coating was evaluated based on color, gloss, reflectance, emissivity, film integrity and adhesion.

Primer formulation No. AF-P14 consisting of the same resin as used in the AF-66 topcoat (SR-123) and pigmented with zinc molybdate at a pigment/binder ratio of 100/100 will provide excellent adhesion, color stability and corrosion resistance.

CONCLUSIONS

Topcoat formulation No. AF-66, when applied over primer formula No. AF-P14, will provide satisfactory service for fifty hours at 700°F. At temperatures of 600°F or less this coating system should perform indefinitely. The reflectance of this formulation after 100 hours exposure at 650°F temperature was 82 percent with 60° gloss exceeding 80 units.

Results of the fuel resistance evaluation indicate that all formulations which contained the General Electric SR-123 resin were more susceptible to softening than were those containing the Dow Corning DC-808 resin when immersed in synthetic lubricant and tricresylphosphate. All formulations evaluated except AF-58-5 (which discolored in Type II fluid) would be satisfactory for field service where the temperature did not exceed 500°F. Thirty minutes at 460°F temperature after fuel immersion, the coatings compounded from both resins regained their original pencil hardness.

Extended temperature exposure for 1000 hours at 460°F did not produce any coating failures. The reflectances of all formulations varied from 80-85 percent and gave 60° gloss readings between 69 and 83 units.

All coatings were satisfactory on all substrates after 500 hours exposure to accelerated weathering.

The coatings applied to 13V11Cr3Al Titanium, 6Al4V Titanium and 301 stainless steel substrates were satisfactory after 1000 hours exposure to a 5 percent salt fog.

The calculated spectral emittance at 80°F and 150°F for formulation No. AF-66 which was representative for all white coatings evaluated was 80 and 81 percent respectively.

None of the coatings subjected to two years of Florida climatic exposure showed any failures other than a negligible decrease in 60° gloss. All coating formulations maintained or increased their reflectance and coating integrity.

SECTION III

FORMULATION INVESTIGATION

A. SELECTION OF RESIN

Because of the higher temperature requirements the selection of resins was limited to the pure silicones. Pure silicones fill the gap between the organic and the inorganic finishes. The properties of the silicone resins that are distinctive are: (1) heat resistance; (2) water repellency; (3) resistance to temperature extremes; (4) excellent weathering and (5) inertness to chemicals and oils after heat cure.

There are a wide variety of silicone modified organic resins that render long service for temperatures that extend from 400°F to 700°F. These organic resins may be added to the silicone resin by either cold blending or by "cooking". As far as temperature resistance is concerned, the listing in Table I may provide a useful comparison.

TABLE I

General Temperature Limitations of Resins

Type of Coating	Maximum Temperature of Usefulness
Organic	150°C (302°F)
Silicone and Organic (cold blend)	200°C (392°F)
Silicone and Organic ("cooked")	200°C (392°F)
Silicone	250°C (482°F)
Silicone + aluminum pigment	500°C (932°F)

These temperatures are intended to be comparative rather than specific depending on the exposures to which they are subjected.

Based on previous research conducted within the Elastomers and Coatings Branch, Nonmetallic Materials Division, the silicone resin which indicated the most merit was a pure silicone manufactured by Dow Corning. This resin, DC-806A, when exposed to a combination of high vacuum and elevated temperature (600°F), gave good color retention and film properties. For this reason the initial research was limited to the DC-806A resin.

B. PROCEDURE FOR REFLECTANCE MEASUREMENTS

Absolute spectral reflectance can be obtained by determining for each spectral (wavelength) band the ratio of radiant energy reflected from a surface to that incident upon it. The optical system must be so arranged to measure directly the incident and reflected components. Measurement of absolute reflectance, consequently, requires accurately calibrated standard sources and reliable, unvarying detectors.

For a comparative evaluation of paints as covered in this report, it is generally sufficient and acceptable to measure reflectance relative to some standard reflector, which has high absolute reflectance throughout wave length region of interest. All reflectance values in this report are of a relative nature.

The reflectance in the visible spectrum (0.4 to 0.7 micron) was measured with a Bausch and Lomb Spectronic 505 Recording Spectrophotometer. The values obtained on this instrument are measured relative to a magnesium carbonate standard and are reproducible within ±0.5 percent.

According to Renford, Schwarz and Lloyd of the General Electric Research Laboratory, magnesium carbonate has an absolute reflectance, which varies from 97 percent at 0.4 micron to 99 percent at 0.7 micron. Magnesium oxide in the same wavelength range, they report, has an absolute reflectance varying from 98 to 99 percent.

To obtain an average value, the ratio was determined for several wavelengths over the entire region. Five points were taken from .40 to .42 microns and averaged and an additional fifteen points taken from .43 to .7 microns. The average value of these are referred to as the total integrated spectral reflectance of the visible spectrum.

C. PREPARATION OF PANELS

All test panels used throughout the initial evaluation consisted of aluminum clad aluminum alloy. The panels were first solvent cleaned with methyl ethyl ketone then treated with a solution of alcoholic phosphoric acid not exceeding two [illegible]tes. The panels were then rinsed with distilled water and the excess water blown off by air.

D. APPLICATION OF COATINGS AND FILM THICKNESS

All coatings were applied by spray application unless otherwise indicated. Previous research conducted on a similar problem revealed that a dry film thickness of a minimum of 3.0 mils would provide sufficient film thickness for reflectance measurements. Any stray light penetrating through this film thickness to the metal substrate would be negligible.

E. PIGMENTATION

The pigmentation of silicone coatings is relatively conventional and with the proper selection of pigment, drier and extender pigment a glossy, highly reflective, heat resistant coating is possible.

Among the white pigments titanium dioxide is the first choice for the same reasons it is of prime importance in conventional coatings. In addition, it has excellent heat stability. There are many types and grades of titanium dioxide on the market today. In general, these are broken down to rutile and anatase. For this particular research the rutile was chosen because of its extremely high refractive index (2.76), superior chalk-resistance, superior hiding power and has less tendency to yellow upon heat exposure at the temperatures involved.

Since reflectance was a major requirement past experience has shown the rutile pigment is superior over the anatase even at low film thicknesses.

A review of various types and grades of rutile titanium dioxide commercially available indicated that R-900 manufactured by E. I. duPont would be one of the better pigments.

The amount of pigment by volume contained in a formulation is generally referred to as the pigment volume concentration (PVC). All the coatings referenced in this report are air dry coatings (room temperature cure). However, upon exposure to heat they develop their maximum physical properties and will be classed as baked-on coatings. For this reason the pigment content will be expressed as pigment to binder ratio by weight hereafter referred to as P/B ratio. A P/B ratio of 0.9 - 1.0 is equivalent to approximately 20 - 22 % PVC.

Driers and antisettling agents were used only in the earlier formulations. In order to minimize the variables which might have an influence on the discoloration which was encountered, they were omitted in the later experimental formulations.

F. EXPERIMENTAL TOPCOAT FORMULATIONS AND THEIR EVALUATION

The primary purpose of this research was to formulate a highly reflective white coating stable for extended time periods at temperatures up to 600°F. Therefore, initial emphasis was placed on the reflectance and other coating properties were of secondary importance. Unless a formulation had a high reflectance before and after heat exposure it was discarded. On this basis all preliminary evaluations were based on reflectance measurements alone. All reflectance measurements were conducted in accordance with Section III. B.

As a result of the literature survey conducted on previous contractual efforts on high temperature coatings, there were several formulations, which have been pigmented with aluminum powder, which would merit evaluation when pigmented with titanium dioxide. The better of these resins was a combination of epoxies and Versamid 115 in various ratios.

In order to have some indication of the heat resistance and color stability of several conventional coatings currently available, a (1) Specification MIL-C-27227 Polyurethane, (2) Specification MIL-L-19537 Acrylic Nitrocellulose and (3) an epoxy, were applied to panels for initial evaluation in 460°F for various time periods. Results of color change are shown in Figure 1. This reflectance curve is representative of the polyurethane and the epoxy coating. The total integrated reflectance initially was quite good, (90%), but dropped to 72% after one hour exposure. Continued exposure decreased the reflectance until at one hundred hours the reflectance was 26%. After this exposure there remained little, if any, vehicle as evidenced by the chalky surface. The acrylic nitrocellulose was extremely thermoplastic as evidenced by bubbling and blistering after thirty minutes exposure to temperature. The color changed to a light tan with a very rough "alligator-skin" surface making a reflectance measurement difficult. The reflectance of the acrylic nitrocellulose was similar to the five hours exposure curve in Figure 1. Continued exposure to one hour merely darkened the coating.

Formulations AF-1 thru AF-6 were cold blends of a pure silicone, Dow Corning 806A, and various other resins consisting of alkyds, acrylics, urethanes, epoxies, and modified urethanes. This series of formulations incorporated the better blending resins available and included the resins which indicated merit from previous Air Force contractual research.

On exposure to temperature, formulation AF-1 after ten minutes exposure had small pin point blisters along the panel edges. There was considerable smoke in the oven presumably from the alkyd portion of the vehicle. Since this formulation was pigmented aluminum, no reflectance curve was run. Formulation AF-2 was a combination of a pure silicone

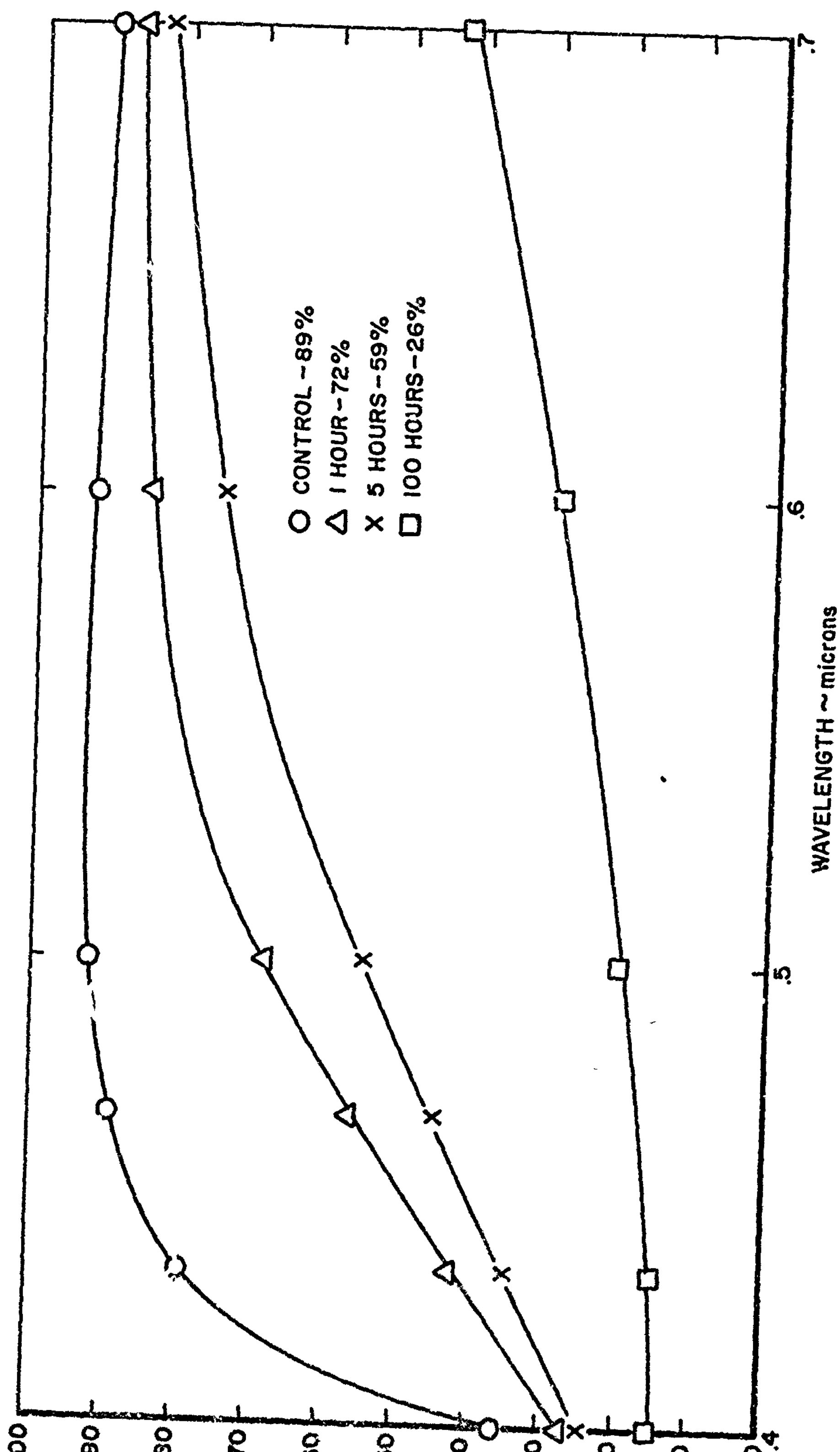

FIGURE 1 - The Reflectance of Specification MIL-C-27227 Before and After 100 Hours Exposure at 460°F

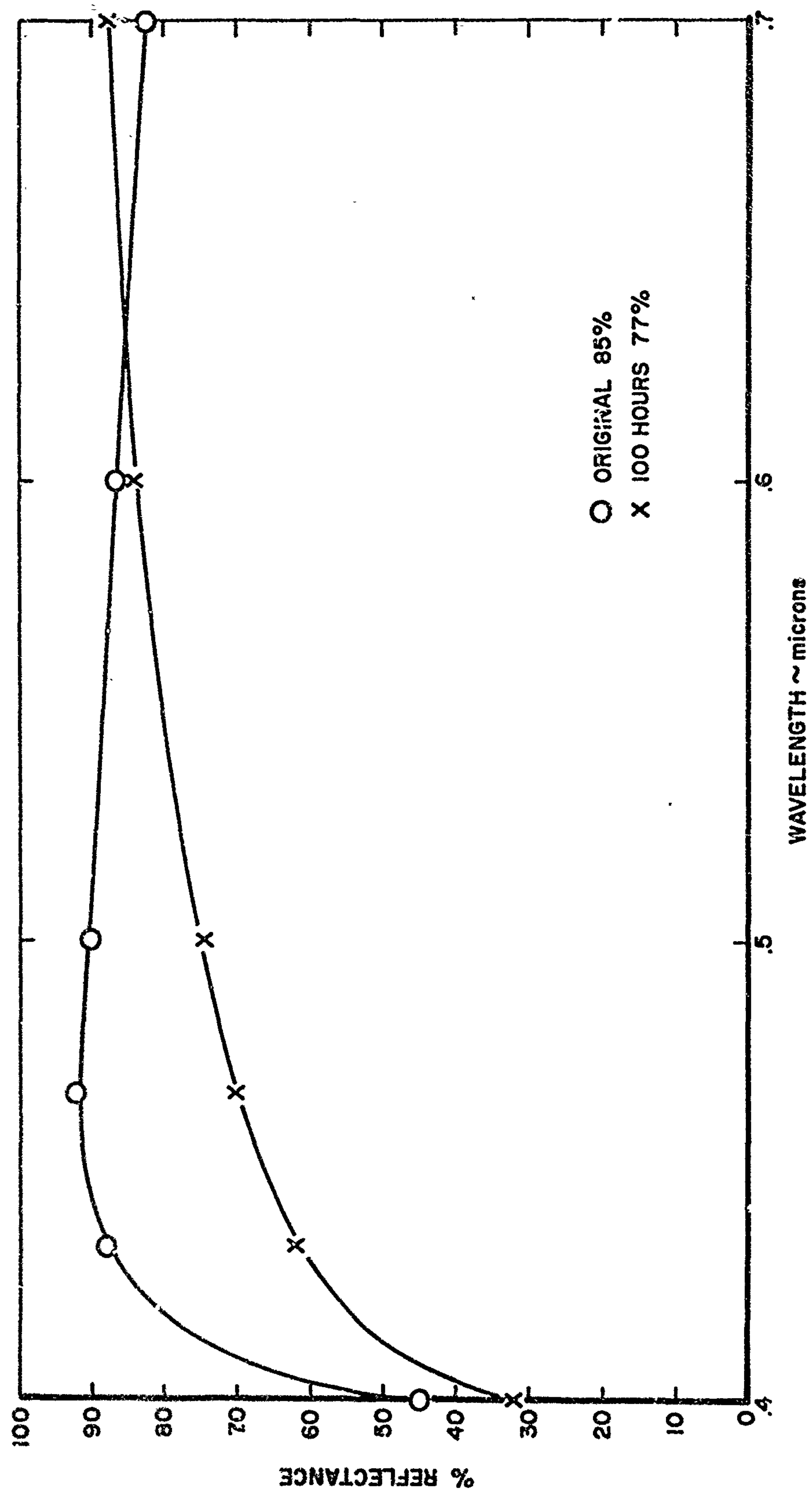

FIGURE 2 - The Reflectance of Formulation No. AF-2B Before and After 100 Hours Exposure at 460°F

DC-806A and an acrylic (A-101) blended at a ratio of 3:1 on resin solids. The surface drying properties of this combination were excellent, but had one serious drawback in that the coating never dried completely hard. Since the large percentage of the resin was a pure silicone, this could be expected. The original reflectance of this coating was 93%, dropping after 5 hours temperature exposure to 77%. Formulation AF-2 was modified by changing the P/B ratio from 100/100 to 50/100 for AF-2A and 25/100 for AF-2B. The lower P/B ratio of formulation AF-2B resulted in the better of the three coatings as can be seen from the reflectance data given in Figure 2. Formulations AF-3, AF-4 and AF-5 consisted of a silicone modified urethane, silicone epoxy and a silicone modified polyester, respectively. After five hours exposure all three formulations turned a very deep brown and had a very chalky surface due to volatilization of the vehicle. The reflectance curve of these samples were very similar to five hours exposure curve listed in Figure 1. Formulation AF-6 consisted of a silicon-alkyd with good air dry properties. This resin was pigmented at a P/B ratio of 60/100. A five-hour heat exposure resulted in a very dark brown coating similar to formulation numbers AF-3, AF-4 and AF-5. The coating did, however, retain some gloss.

Formulation AF-7 was compounded to see the effects of discoloration on a pure silicone. Results of the color retention were excellent.

Formulation AF-8 consisted of combination of silicone and acrylic at a 4:1 resin solids and a P/B ratio of 45/100. This semi-gloss coating had a reflectance of approximately 85% after five hours heat exposure. The actual reflectance was not measured due to instrument breakdown. After continued heat exposure thru one hundred hours the acrylic portion of the resin combination had turned to a light beige. The reflectance after one hundred hours was approximately 80%. The surface was very chalky and little gloss remained. The air dry properties were not as good as expected using the zinc octoate and the coating was very brittle.

Formulation AF-9 was the same as formulation AF-8 only the zinc octoate was replaced with cobalt naphthenate. Results were the same as in formulation AF-8.

Formulation AF-10 consisted solely of a clear acrylic exposed to temperature to determine its suitability as a blending resin for the pure silicone. A five hour heat exposure showed the resin to volatilize and discolor rendering it unsatisfactory. Formulation AF-11 was a pure silicone compounded by Lowe Bros. for another project and coded LH-2370. This coating was extremely "flat". Exposure to temperature for five hours did increase the gloss slightly but yellowed the coating. Formula-

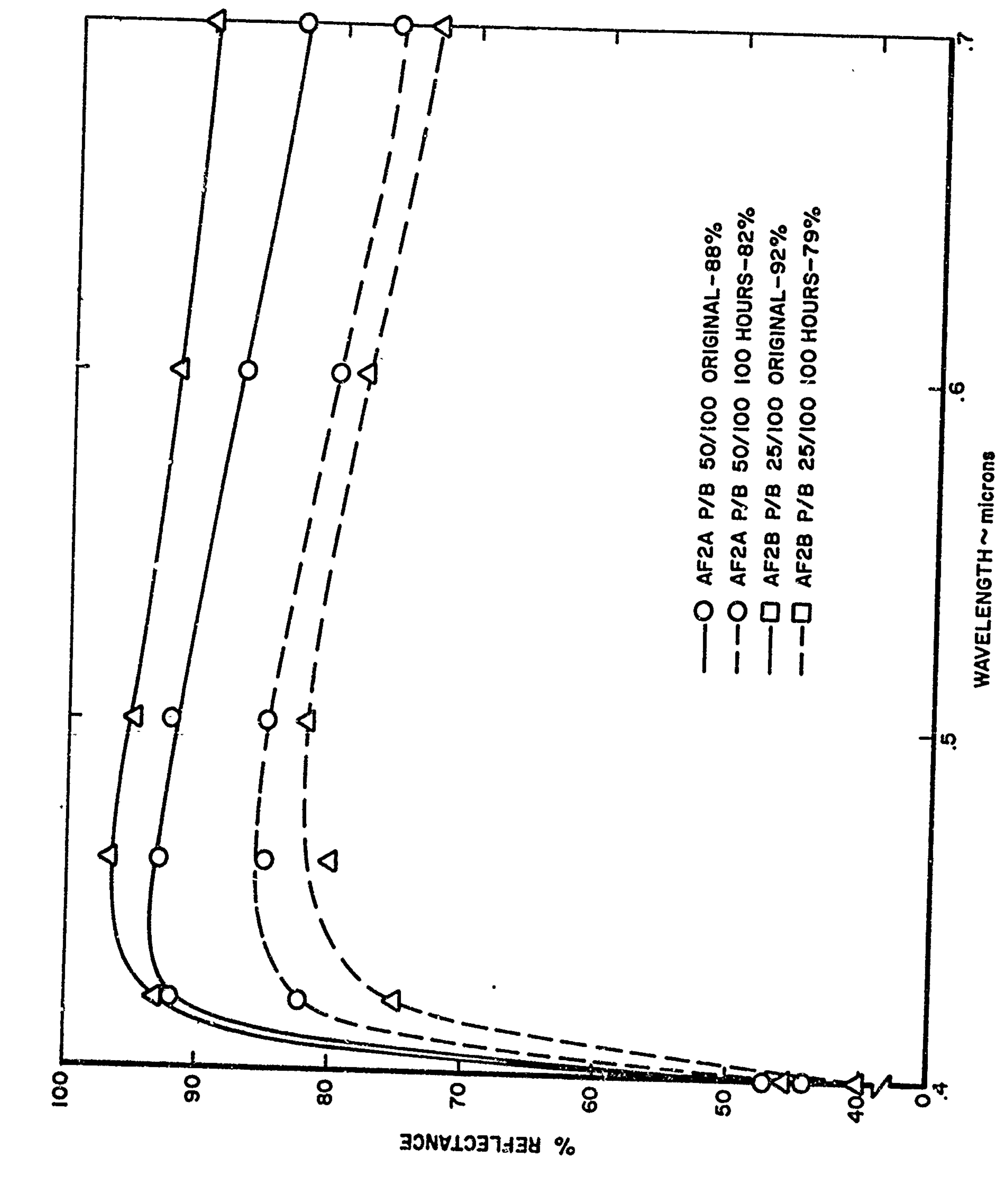

FIGURE 3 - The Effect of P/B Ratio on Reflectance of Formulation No. AF-2A and AF-2B Before and After 100 Hours Exposure at 460°F

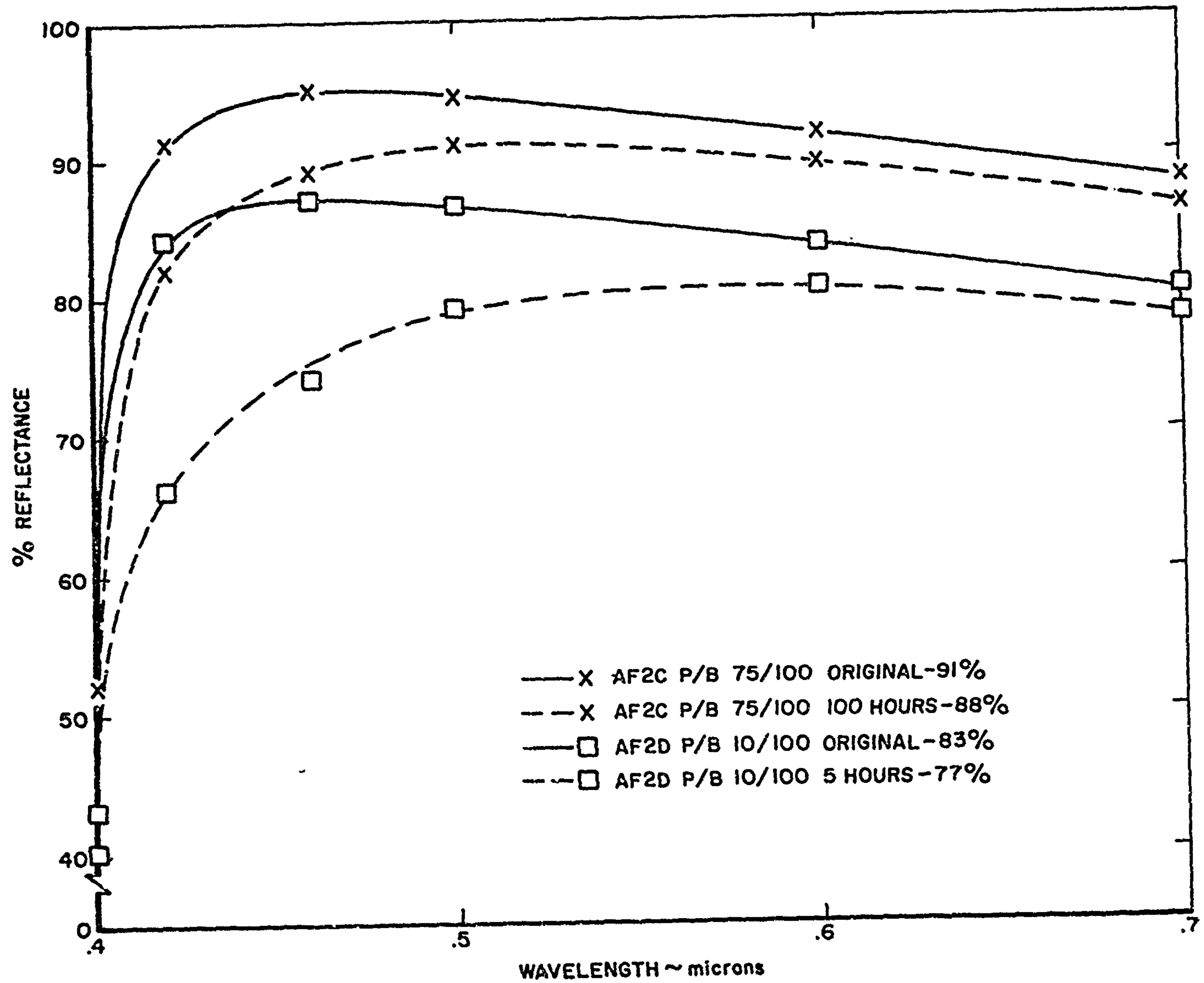

FIGURE 4 - The Effect of P/B Ratio on Reflectance of Formulations AF-2C and AF-2D Before and After 100 Hours Exposure at 460°F

tion AF-2A, AF-2B, AF-2C and AF-2D are a series of modifications of AF-2 which gave the excellent color retention as can be seen in Figure 3 and 4. In all these formulations the silicone (DC-806A), and the acrylic (A-101) resin ratio was held at the level of 3.7:1 on resin solids. The only variation was the pigment to binder ratio. This ratio varied from 10/100, 25/100, 50/100 and 75/100. Of this series of formulations, AF-2B and AF-2C had the highest initial reflectance. After 100 hours exposure AF-2B dropped to 79% in reflectance whereas AF-2C had a 3% decrease in reflectance. Since AF-2C was the highest P/B ratio, it appears that the increased pigment content is a determining factor in the reflectance stability of the coating. Figures 3 and 4 show the effect of P/B ratio on reflectance. Formulations AF-11, AF-12, AF-13, and AF-14 were formulated with the hope that individually the urethanes would not discolor as severely as when combined. However, after one hour's exposure to temperatures, the reflectance had dropped severely and was representative of the one hour exposure curve shown in Figure 1. Formulations AF-15 and AF-16 were blends of a silicone-alkyd and an acrylic. This combination would not air dry. After five minutes heat exposure, there was a severe decrease in reflectance accompanied by surface blistering. Formulation AF-17 was formulated with the silicone acrylic based on the results obtained from formulations AF-2A through AF-2D. The P/B ratio was increased to 100/100. The reflectance both originally and after one hundred hours exposure was essentially the same as formulation AF-2C. There was a slight loss of gloss after exposure. The film was very brittle and could easily be flasked off by the thumb nail.

Formulations AF-18, AF-19, AF-20 and AF-21 are modifications of formulation AF-2 varying the silicone/acrylic ratio from 1:1 to 1:2 based on resin solids. The P/B ratio was held at two levels of 25/100 and 50/100.

In comparing these four formulations, the reflectance of AF-18 was the poorest as shown by a drop in reflectance from 82% to 62% after 100 hours. Although the coating was a light tan there still remained a fair amount of gloss. Formulation AF-19 was originally a semi-gloss coating. After exposure to temperature for thirty minutes the coating was 100% covered with tiny blisters. The gloss decreased to a flat finish. Since there was no discoloration, this coating was exposed for 100 hours. Although there were no additional blisters the reflectance dropped from the original of 89% to 79%. The adhesion after exposure was excellent in comparison to the extremely brittle character prior to heat exposure. Formulation AF-20 was very similar to AF-19 in that it was brittle and blistered after thirty minutes heat exposure. After thirty minutes heat exposure the reflectances dropped to 91%. Because

of the severe blistering and since this formulation was very similar to AF-18 in resin combination, there was no additional heat exposure. Formulation AF-21 had an original reflectance of 93% which decreased to 87% after one hundred hours heat exposure. The only blistering encountered was at the end of the panel where the coating was heavier in film thickness. There was a slight increase in gloss after heat exposure resulting in a semi-gloss coating. In general, this formulation was the best of this series.

Formulation AF-22 was a combination of AF-18 and AF-20 in that the resin ratio was held at 1:1 and the P/B ratio increased from 55/100 to 100/100. It was thought that by increasing the P/B ratio and the addition of a slower medium boiling solvent such as methyl isobutyl ketone in lieu of the toluene, one would obtain a dry film with less trapped solvent thereby reducing the severe blistering encountered in AF-18 and AF-20. Results however were the same; severe blistering resulted at the end of thirty minutes heat exposure. Continued exposure produced no additional blistering and only a slight decrease in reflectance. The original reflectance was 86% decreasing to 82% after thirty minutes exposure and a further slight decrease to 79% after 100 hours heat exposure.

Formulations AF-23 through AF-27 were compounded to determine what ratio of silicone to acrylic would be most desirable with various P/B ratios. Formulations AF-23, AF-24, AF-25, AF-26 and AF-27 all had blistering of various degrees. The blisters were few and isolated but present. Formulation AF-27 appeared the whitest as shown by the extremely high reflectance of 95%. After 50 hours heat exposure the reflectance dropped 89%. This 6% decrease in reflectance was characteristic of the decrease in reflectance of the other formulations which varied in original reflectance from 86% to 93% then decreased to 79% after heat exposure. The adhesion of formulations AF-23 through AF-26 varied from good to poor. Formulation AF-27 had very good air dry adhesion while AF-26 had the fewest number of blisters undoubtedly due to the highest P/B ratio of this series. It appears that AF-26 pigmented at a 50/100 P/B ratio and a 9:1 vehicle ratio would provide the better coating.

Formulation AF-28 was based on the above data. It was pigmented at 75/100 P/B ratio with a 9:1 vehicle ratio using DC-806A silicone and A-101 acrylic. This formulation resulted in very poor application properties. The original reflectance was 87%. After three hours heat exposure the coating was so severely blistered the reflectance could not be determined.

Formulations AF-29 through AF-33 were formulated with the P/B ratio varying from 33/100 through 150/100 and holding the silicone-acrylic

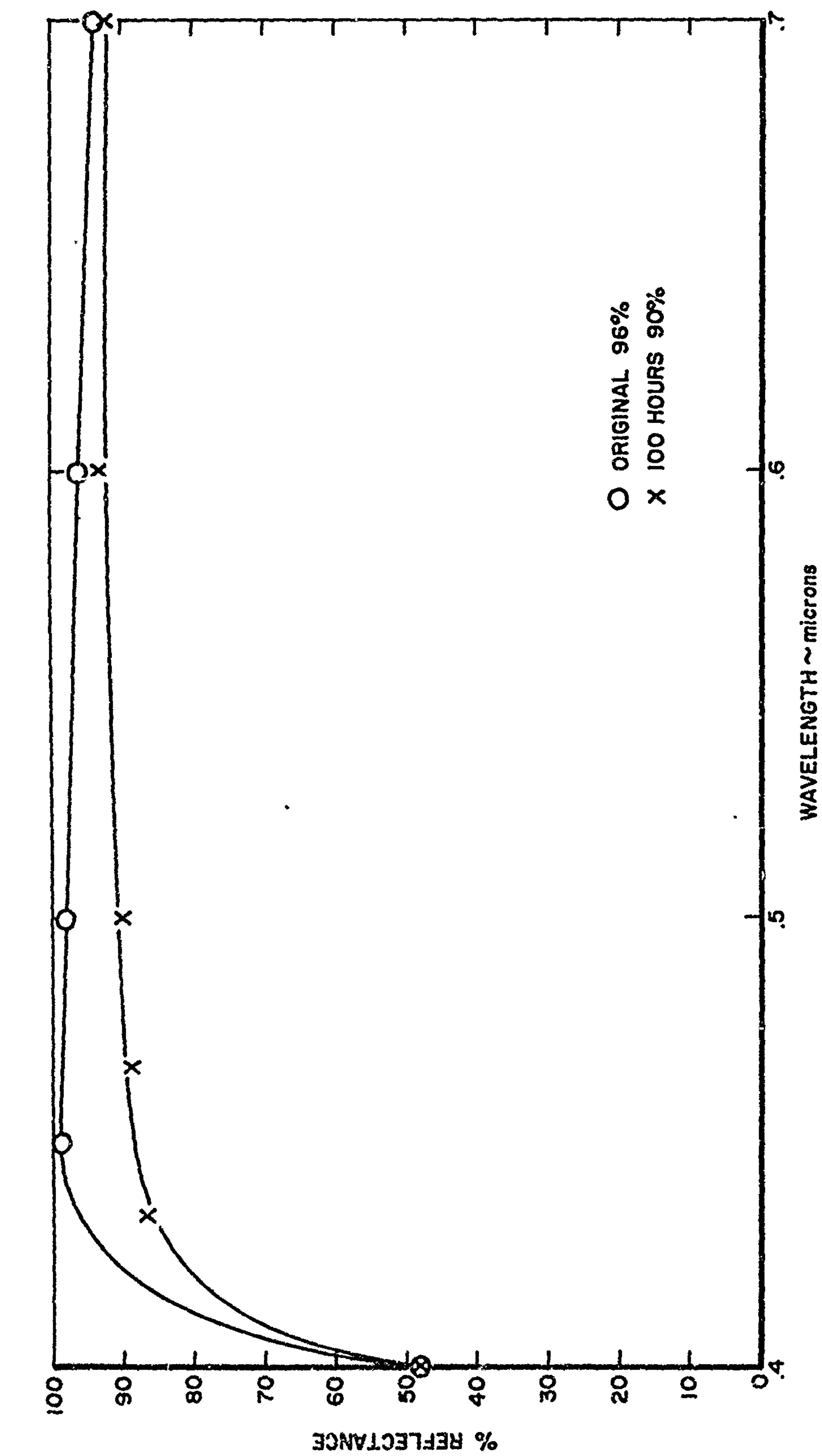

FIGURE 5 - The Reflectance of Formulation AF-32 Before and After 100 Hours Exposure ; 460°F

ratio constant at 2.4:1. By increasing the acrylic ratio, it was anticipated that the residual solvent trapped within the silicone would be minimized, thereby eliminating the severe blistering previously encountered. Formulations AF-30 and AF-31 were both so severely blistered at the end of 100 hours heat exposure that the reflectance values obtained are questionable. The original reflectances of these coatings were 96% and 97% and they decreased to 85% and 89% respectively. Formulation AF-29 had very small scattered blisters covering the entire panel. The reflectance decreased from 98% to 90% after 100 hours heat exposure. Formulation AF-32 showed no evidence of blistering. This is unexplainable since the P/B ratio was very nearly the same as formulation AF-29. It is not likely the 10% decrease in pigment would have such a great effect. The film thickness of both formulations AF-29 and AF-32 was the same (2 mil) as were the drying times. Although the reflectance of AF-32 was not as high as AF-29, the original reflectance of 96% decreased to 90% after 100 hours heat exposure as can be seen in Figure 5. Formulation AF-33, which had the highest pigment loading did not blister. The original reflectance of 97% decreased to 85% after 100 hours heat exposure. The fact that this coating did not blister, retained some gloss and maintained a very good reflectance made this formulation one of the better ones to date.

Formulations AF-34 and AF-35 were formulated based on the results of AF-33. The silicone/acrylic ratio was held at 2.4:1 with the P/B ratio increased from 150/100 to 200/100 and 300/100.

Formulation AF-34 resulted in a semi-gloss coating that had fair adhesion but was slightly brittle. The original reflectance of 90% dropped to 67% after 100 hours exposure to heat. Formulation AF-35 was flatter than was AF-34 but still not a true flat. The original reflectance of 94% decreased to 90% after one hour heat exposure then to 81% after 100 hours exposure. Therefore it appears that the optimum P/B ratio is somewhere between 90/100 and 150/100 depending upon the type of coating desired.

Formulation AF-36 was formulated using a proprietary experimental polymer of the polyester type. This polymer was pigmented at a 100/100 P/B ratio. The original reflectance of 93% decreased to 52% after thirty minutes heat exposure, then fell to 10% after an additional twenty-four hours heat exposure. No additional research was conducted on this polymer.

Formulation AF-37 was a 1:1 by resin solids cold blend of DC 806A and DC 805 silicones. This in turn was blended with the A-101 acrylic at a ratio of 3:1 on resin solids. The resin was then pigmented at 25/100 P/B ratio.

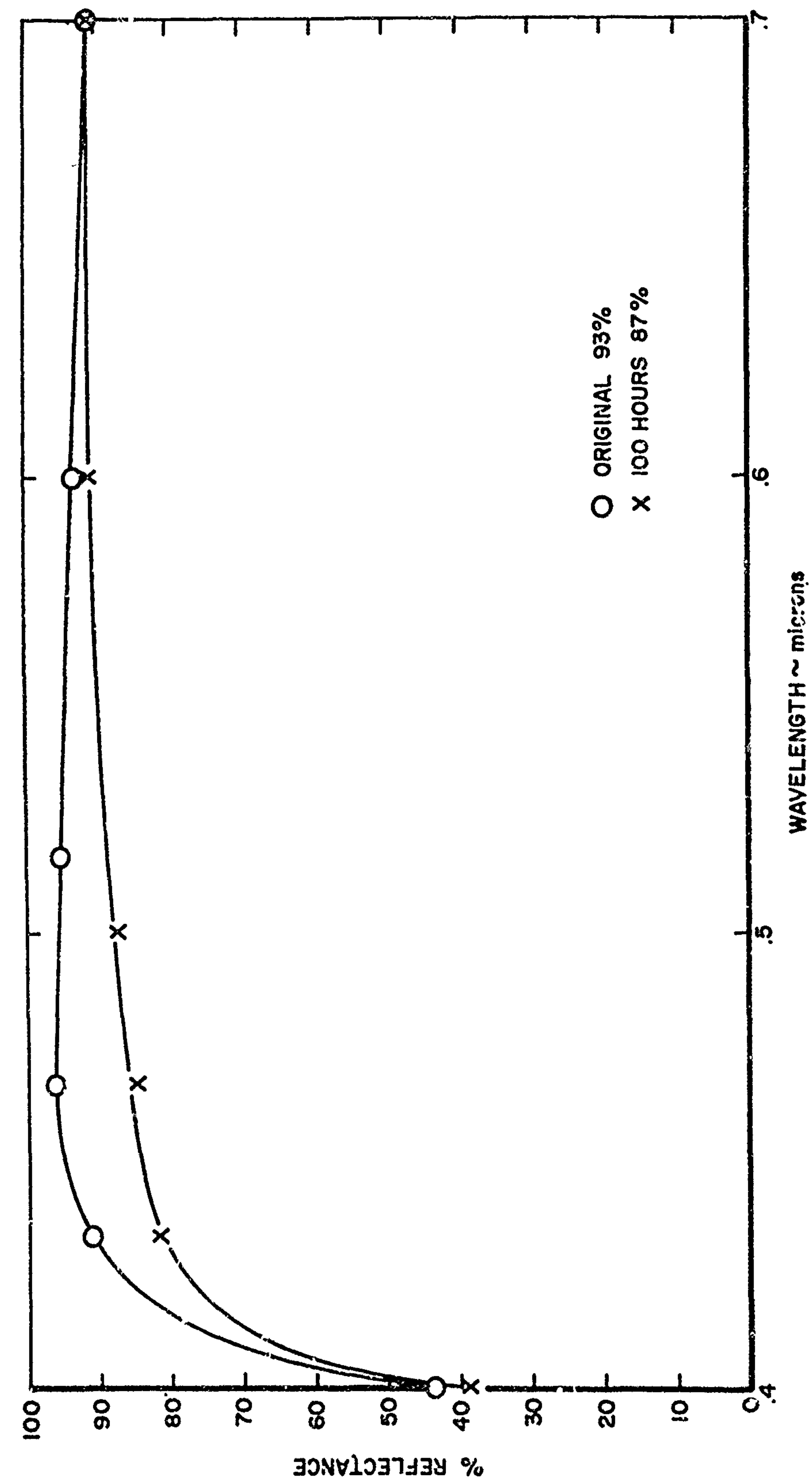

FIGURE 6 - The Reflectance of Formulation No. AF-41 Before and After 100 Hours Exposure at 460°F

During a review of past formulations and examination of the coated panels, it was found that many of the panels had "crowfooting" to various degrees. Further examination of additional older panels revealed that the "crowfooting" had developed into "alligatoring". Although the cracking had not penetrated to the base substrate, it destroyed the usefulness of the coating. DC 806A silicone is a relatively hard resin and this defect had not been previously encountered during any evaluation. It was decided that in order to increase the flexibility of DC 806A, to cold blend this resin with DC 805 which is a much more flexible resin. Although this resin does not have the vacuum stability of the DC 806A, it is compatible with DC 806A and in addition the viscosity, color, service temperature and solids are essentially the same. Better still, the weathering resistance and flexibility are twice that of DC 806A. The other possible reason for this cracking is that the titanium dioxide pigment was reacting with the silicone vehicle.

Formulations AF-38 and AF-39 were formulated using two extremely flexible silicone alkyd resins reportedly having very good heat stability. AF-38 was pigmented at 25/100 and AF-39 at 100/100 P/B ratio. Both of the formulations had excellent flexibility.

The original reflectances of AF-38 was 88% but after one hour's heat exposure dropped to 50%. Formulation AF-39 had an initial reflectance of 93%. After one hundred hours heat exposure this coating still maintained a reflectance exceeding 88%. Coupled with its good flexibility this formulation merits further consideration and evaluation.

Formulation AF-40 was formulated using DC 806A and A-101 acrylic. This acrylic is an extremely hard resin and is generally used for heat resistant white enamels. The P/B ratio was held at 50/100 with the silicone/acrylic ratio at 3:1. The initial reflectance of 90% decreased to 81% after one hour's heat exposure. In addition to the blistering, the coating had little or no adhesion.

Formulations AF-41, AF-42 and AF-43 were formulated in an attempt to increase the flexibility and eliminate the cracking previously reported in Formulation 2B. The P/B ratio was held at 100/100 for all formulations. Formulation AF-41 was DC 806A silicone and A-101 acrylic pigmented with titanium dioxide. In formulation AF-42, 5% of the titanium dioxide used in formulation AF-41 was replaced with mica. Formulation AF-43 was straight DC 806A silicone and pigmented the same as formulation AF-42. AF-41 resulted in a semi-gloss coating with an initial reflectance of 92%. After 100 hours heat exposure, the reflectance was 87% as can be seen in Figure 6 with no loss of gloss. There were, however, very fine hairline cracks covering the entire panel. Thirty days after application the air dried panel also had hairline cracks.

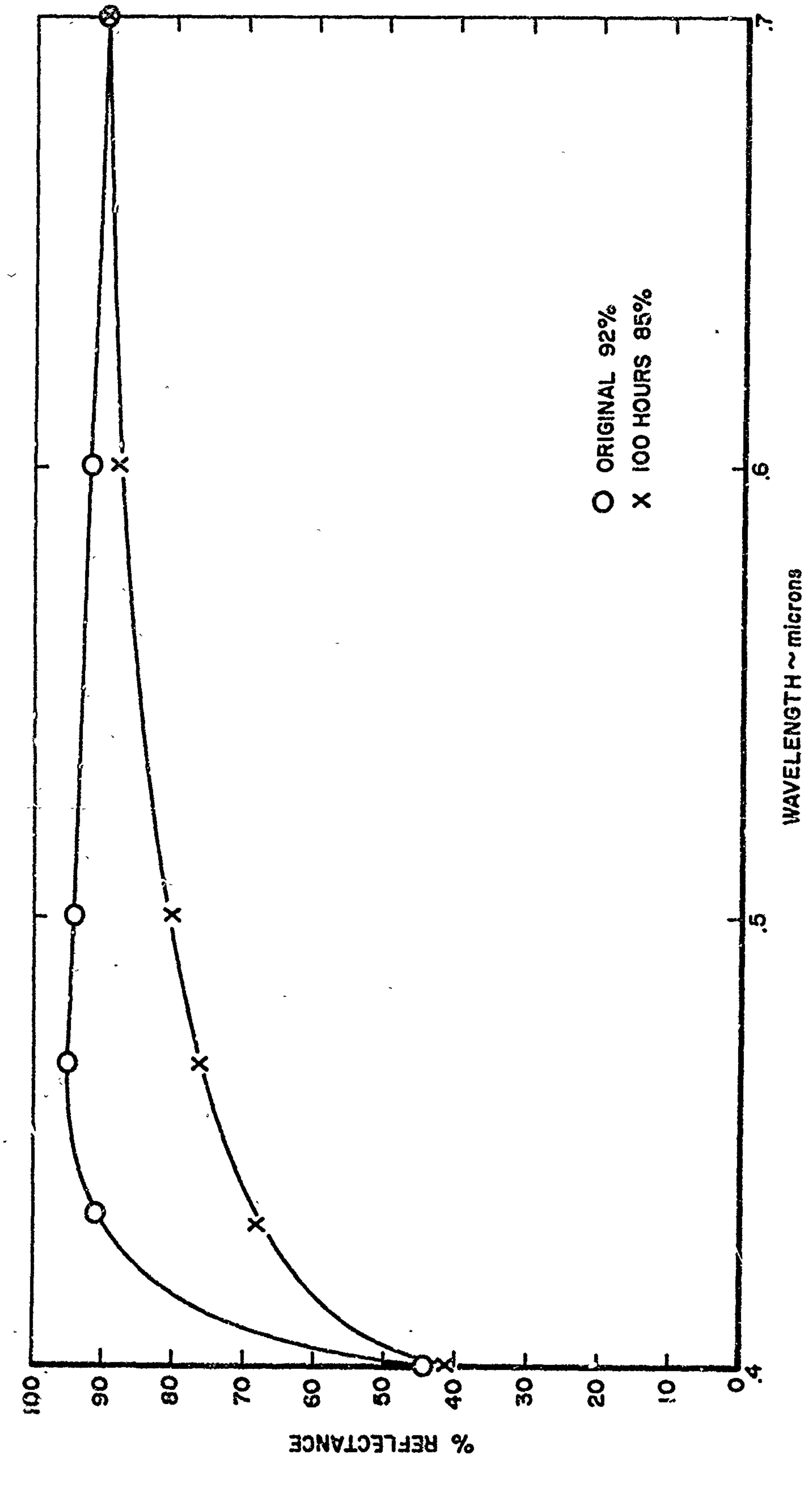

FIGURE 7 - The Reflectance of Formulation No. AF-45 Before and After 100 Hours Exposure at 460°F

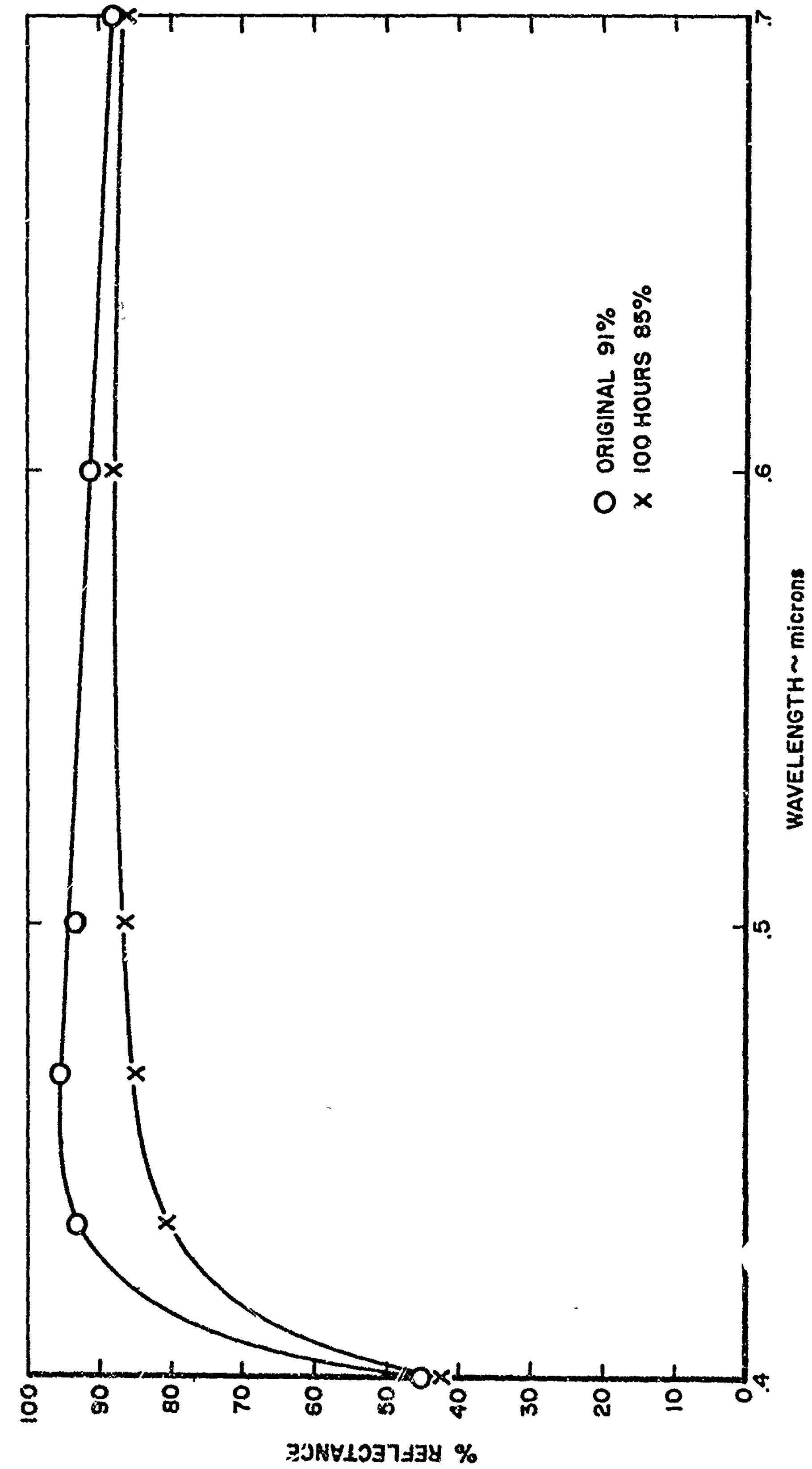

FIGURE 8 - The Reflectance of Formulation AF-47 Before and After 100 Hours Exposure at $460^{o}F$

After further investigation into the severe cracking encountered, it was believed that substitution of RA-10 or RA-NC might alleviate the situation. At this time a sample of Dow Corning amino-functional silane (Z-6020) was considered for potential use as a drying agent. This material was known to have the ability to couple various organic and inorganic materials.

Upon receipt of the Z-6020 catalyst a series of coatings were pigmented with R-900 titanium dioxide holding the P/B level at 25/100 and varying only the type of resin. All coatings were catalyzed with Z-6020 at 3% based on vehicle solids. Formulation AF-44 through AF-48 were compounded as follows:

TABLE II

Reflectance Results of Formulations AF-44 through AF-48

Formulation	Resin	Original	100 Hours at 460°F	Impact Adhesion	Pencil Hardness
AF-44	DC-806	90	87	6	F
AF-45	DC-801	92	85	28+	F
AF-46	DC-805	86	79	28+	4B
AF-47	DC-808	91	85	28+	B
AF-48	DC-0031	no cure	--	---	--

Results of the above evaluation indicated that the Z-6020 catalyst did to a certain extent cure the pure silicones. The DC-806 resin had the lowest reflectance drop, but was extremely low in impact resistance. The DC-805 resin was very soft in pencil hardness, had fair reflectance but did have good impact adhesion. The DC-801 and DC-808 resins had good reflectance, impact adhesion and pencil hardness, but the DC-808 resin had a slight advantage over the DC-801 in retention of reflectance. Since formulations AF-45 and AF-47 had such good results as can be seen in Figure 7 and 8, these resins merited further evaluation.

In order to determine if the cause of the severe cracking was a result of the silicone resin reacting with the R-900 pigment, formulations AF-49 and AF-50 were pigmented with RA-50 and FF titanium dioxide in lieu of the R-900.

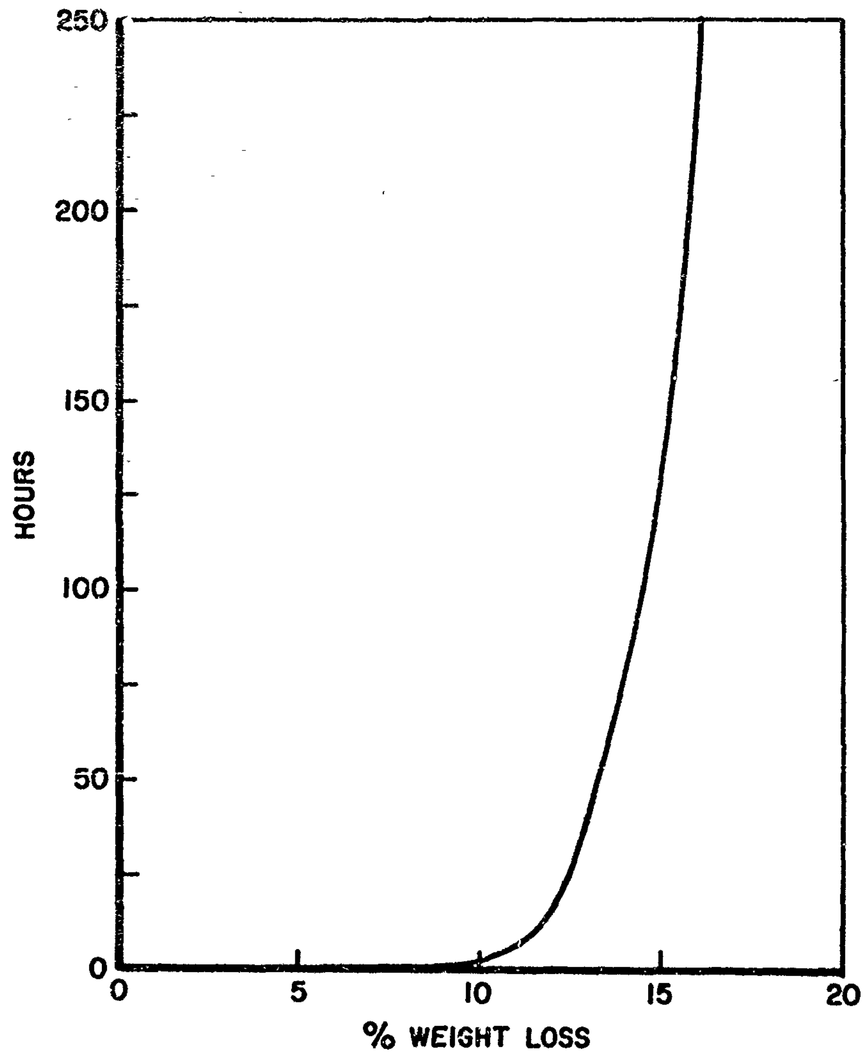

FIGURE 9 - The Percent Weight Loss on the Clear Vehicle of DC-808 Resin at 650°F

Formulations No. AF-49 and AF-50 had initial reflectances of 91%, but decreased to 82% and 80% respectively after one hundred hours heat exposure. After thirty days air dry, the coatings had severe cracking.

As a possible solution for the elimination of the severe cracking encountered, three additional titanium dioxide pigments were requested. These pigments, R-100, R-200 and R-610 were formulated into both DC-806A and DC-808 resins holding the P/B ratio 25/100. The DC-808 resin was chosen because of its exceptionally good flexibility and heat stability as shown in Figure 9. Formulations AF-51, AF-52 and AF-53 were used for DC-806A while AF-54, AF-55 and AF-56 were used for the DC-808. At the end of four hours at temperature, all six formulations had discolored to the point where all tests were terminated. The better of the DC-806A formulations was AF-51 which contained the R-100, while formulations AF-52 and AF-53 showed evidence of cracking. The same three pigments used in the DC-808 resin showed no evidence of cracking but severe discoloration.

The DC-808 when pigments with R-900 titanium dioxide as in formulation AF-47 gave results that were far superior to the R-100, R-200 or the R-610 as shown in Table III.

TABLE III

Reflectance Results of Formulations AF-47, AF-51 through AF-56

Formulation	Resin	Pigment	Original	After Exposure	Hours at 460°F
AF-47 (control)	DC-808	R-900	91	85	100
AF-51	DC-806	R-100	93	77	4
AF-52	DC-806	R-200	88	78	4
AF-53	DC-806	R-610	86	70	4
AF-54	DC-808	R-100	90	76	4
AF-55	DC-803	R-200	92	82	4
AF-56	DC-808	R-610	86	73	4

Based on the manufacturer's information, formulation AF-57 was a combination of G. E. resins which when combined would be equal to DC-806A. A cold blend of these resins when catalyzed with Z-6020 resulted in an incompatible solution as evidenced by two distinct solution layers. Work on this combination was terminated.

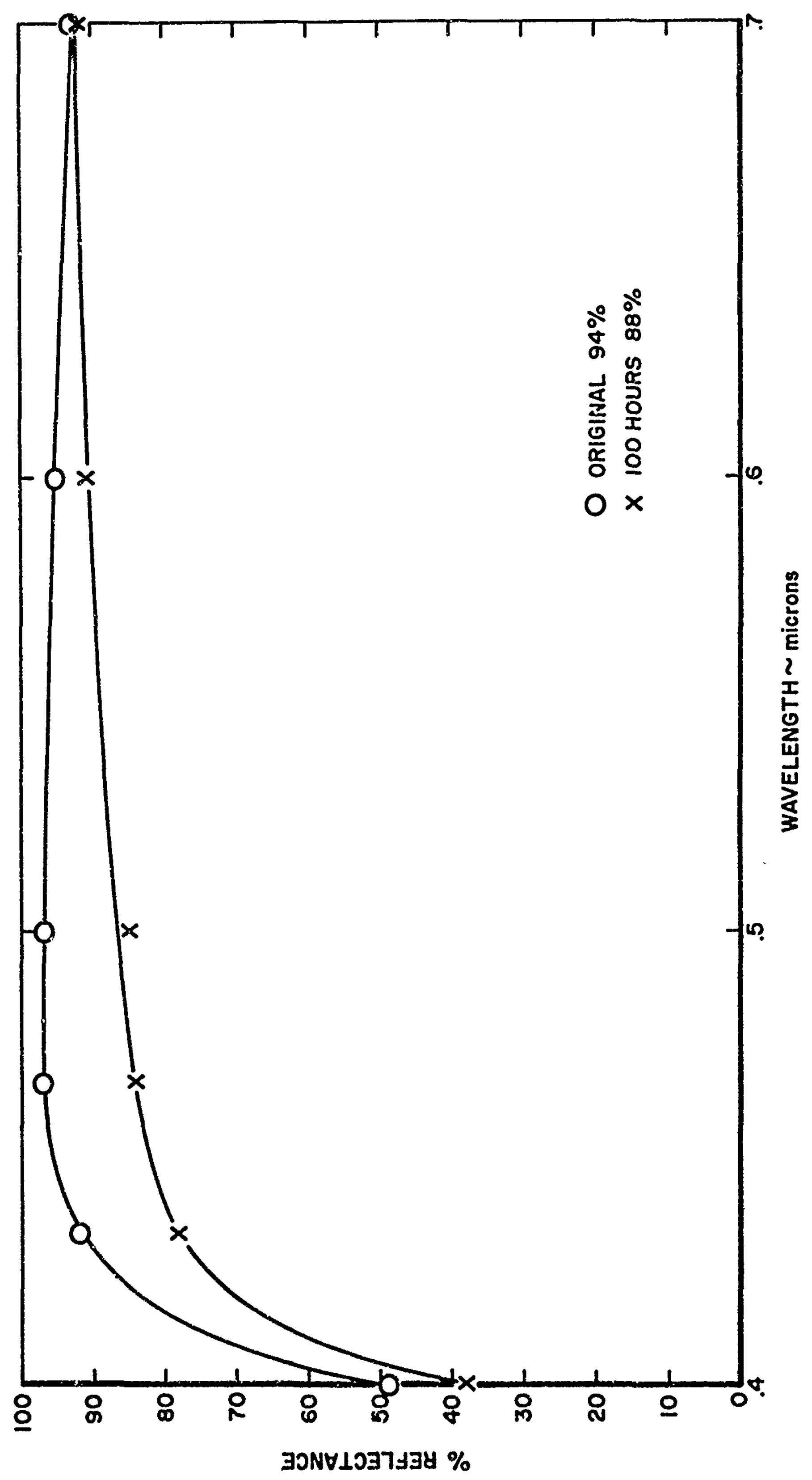

FIGURE 10 - Reflectance of Formulation AF-58 Before and After 100 Hours Exposure at 460°F

Formulation AF-58 was compounded based on the information obtained on formulation AF-47. In order to increase the hiding power a P/B ratio of 50/100 was used in lieu of the 25/100 as in formulation AF-47. Reflectance results were better than anticipated as can be seen in Figure 10, with the original reflectance of 93.5% dropping to 88.4% after 100 hours temperature exposure. Formulation AF-59 was formulated as a possible substitute for titanium dioxide in limited space applications. Zirconium dioxide was incorporated into the vacuum stable DC-806A at a P/B ratio of 50/100. When exposed to temperature for four hours the original reflectance of 79.9% dropped to 56.1%. Further evaluation was terminated.

In order to eliminate the cracking previously reported, a sample of RA-10 titanium dioxide was compounded into formulations AF-60 through AF-65 with P/B ratios of 25/100, 50/100 and 100/100. The coatings were catalyzed with Z-6020 at 3% based on resin solids. The resins used were DC-806A and DC-808 pigmented as shown in Table IV:

TABLE IV

Reflectance Results of Formulations AF-60 through AF-65

Formulation	Resin	P/B	Original	After Exposure	Hours at Temperature
AF-60	DC-806A	25/100	90	82	100
AF-61	DC-806A	50/100	92	86	100
AF-62	DC-806A	100/100	92	83	100
AF-63	DC-808	25/100	90	80	100
AF-64	DC-808	50/100	90	78	100
AF-65	DC-808	100/100	93	83	100

Results of the above formulations were satisfactory for the DC-808 resin but the DC-806A formulations showed cracking after two weeks air dry. The cracking decreased as the pigment volume was increased. There was no evidence of cracking on any of the coatings using the DC-808 resin after an extended air dry. After one hundred hours heat exposure, all three DC-806A formulations showed cracking to various degrees. The DC-808 pigmented resin showed no cracking. The reflectance of all coatings were quite good as indicated in Table IV but still not as high as was AF-58 which had 88% reflectance after one hundred hours temperature exposure.

The DC-808 resin pigmented coating had the greatest decrease in reflectance. There was the possibility that the Z-6020 catalyst was causing some of the discoloration encountered after heat exposure. Therefore,

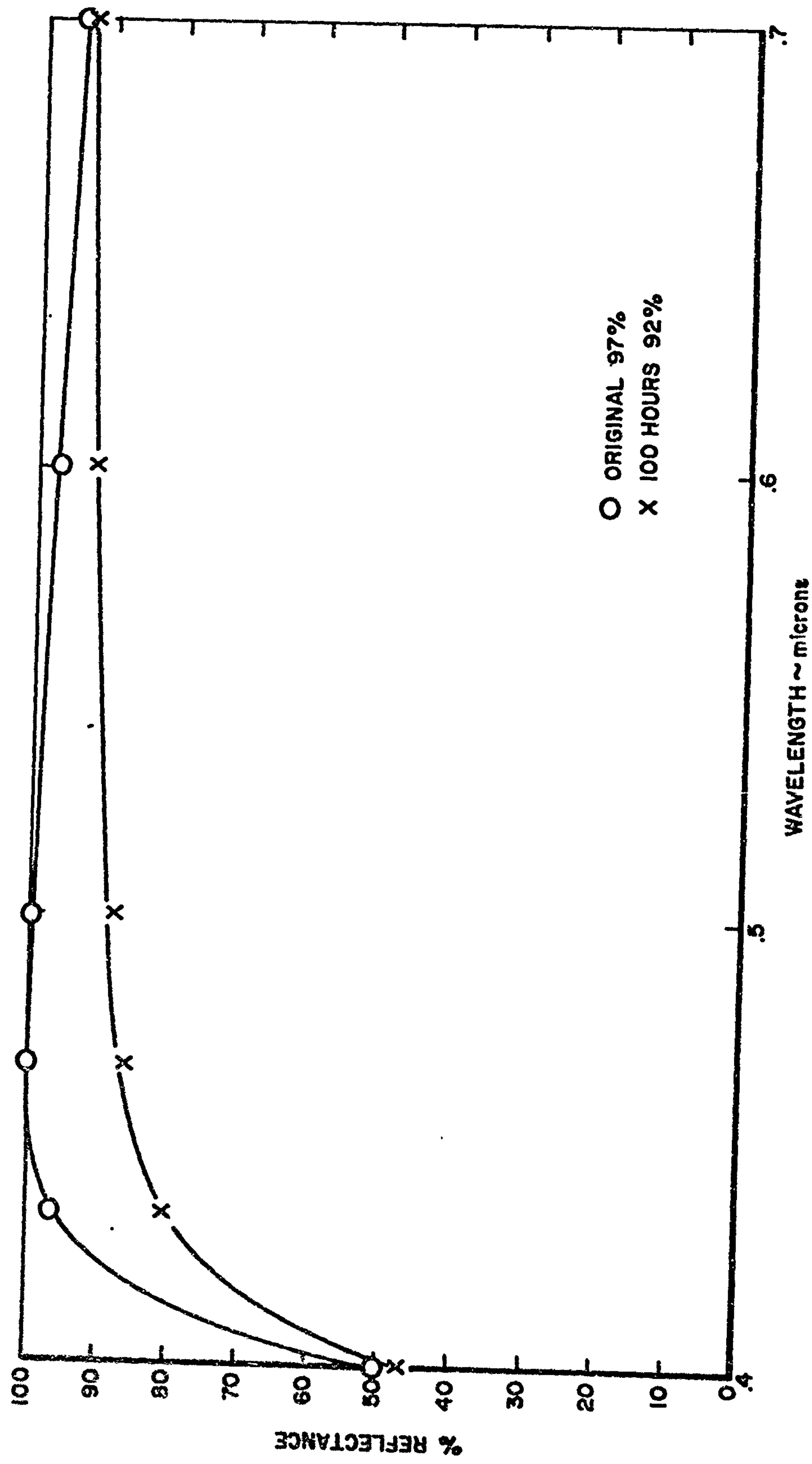

FIGURE 11 - The Reflectance of Formulation AF-58-5 Before and After 100 Hours Exposure at 460°F

formulations AF-60 through AF-65 were again applied, allowed to air dry for forty-eight hours, then heat cured at 300°F for two hours to remove any residual solvent before subjecting them to temperature exposure. As a result of the elimination of the Z-6020 catalyst, all formulations after one hundred hours heat exposure had a very slight (less than 2%) decrease in reflectance. Therefore, it can be assumed that the Z-6020 does decrease the initial reflectance of the pigmented coating.

As a result of the research conducted on the discoloration of white coatings when catalyzed with Z-6020, it was found that:

1. A minimum of 2% catalyst is necessary to promote a satisfactory cure and provide maximum reflectance.

2. The use of the catalyst in excess of 2% results in a severe decrease in reflectance and adds little to the coating.

3. Coatings formulated using DC-808 resin and catalyzed at 2% resulted in an impact adhesion exceeding 26 inch pounds.

In order to improve the weathering properties of formulation AF-58, (modified AF-47), various percentages (5%, 7.5% and 10%) titanium dioxide were replaced with zinc oxide holding the P/B ratio at 100/100. The original reflectance of all three coatings were quite good with no visible decrease in gloss. Reflectance results of formulations AF-58, AF-58-5, AF-58-7.5 and AF-58-10 are given in Table V.

TABLE V

Reflectance Results of Formulations AF-58, AF-58-5, AF-58-7.5 and AF-58-10

Formulation	Pigment	Original	Percent Reflectance 2 hr Exposure	Percent Reflectance 100 hr Exposure
AF-58	R-900	94	87	± 1% No Change
AF-58-5	R-900+5% ZnO	97	92	"
AF-58-7.5	R-900+7.5% ZnO	97	86	"
AF-58-10	R-900+10% ZnO	97	96	"

When formulation AF-58 (Figure 10) is compared with the three formulations containing zinc oxide there is a slight increase in the original reflectance for formulation AF-58-5 (Figure 11). It appears that the optimum of zinc oxide is 5%. This percentage increases the reflectance both

before and after heat exposure. Any additional increase has no material benefit upon reflectance. Additional extenders consisting of zinc sulfide and mica were evaluated but due to the severe loss in ititial gloss the tests were terminated.

Based on the excellent data obtained with the use of Z-6020 as a catalyst, two additional amino silanes (XA-1902) and (A-1100) were evaluated as possible replacements for the Z-6020.

Using formulation AF-58 as the basic coating, selected catalysts were incorporated into the coating at 2, 5, 10 and 15% based on resin solids. Results were that the drying time of the Z-6020 at 2% was a few minutes faster than the XA-1902 or A-1100 at 5 and 10% for obtaining a dust free surface. The XA-1902 and A-1100 at 10% were almost twice as fast for obtaining a tack free surface than was the Z-6020 at 2%. The coating catalyzed with the A-1100 at 10% had a slight advantage in obtaining a tack free surface over the same coating using a 5% catalyst. After forty-eight hours drying time, the coating catalyzed with the A-1100 at 10% resulted in a much harder film than did the XA-1902 at 10% or the Z-6020 at 2%. Since this was a cursory examination there was no attempt to maintain any specific film thickness. All of the above films were applied using an eight mil draw down gage resulting in a wet film thickness of approximately four mils.

In response to a quentionnaire distributed by Southern Research Institute under NASA sponsorships, for a white, high temperature coating capable of withstanding a temperature of 650°F for prolonged periods of time, and to simultaneously determine the drying time in accordance with Federal Standard 141, Formulation AF-58-5 was catalyzed with amines A-1100 and A-1902 at 10% and Z-6020 at 3%. These coatings were applied by draw down to a wet film thickness of 1.5 mils with drying times as indicated in Table VI:

TABLE VI

Drying Time of Catalyzed Coatings

Top Coat	Catalyst	Set to Touch	Dust Free	Tack Free
AF-58-5	A-1100	5 minutes	7 minutes	60 minutes
AF-58-5	XA-1902	5 minutes	7 minutes	60 minutes
AF-58-5	Z-6020	5 minutes	7 minutes	60 minutes

In order to insure complete hiding of the metal substrate and using the same catalyzed material, several panels were prepared by draw down (8 mil wet) using the above systems. The panels were air dried for seventy-two hours and, in lieu of the normal 460°F exposure, were subjected to a temperature of 650°F. Reflectance results were as follows:

TABLE VII

Percent Reflectance after Exposure at 650°F

Top Coat	Catalyst	Original	1 Hr.	2 Hrs.	18 Hrs.	100 Hrs.
AF-58-5	A-1100	94	84	80	86	90
AF-58-5	XA-1902	94	83	74	81	Flaked off
AF-58-5	Z-6020	94	82	74	82	87

Within plus or minus of one percent which is within the instrument error, there was no difference in the original reflectance. All three coatings suffered a severe decrease in reflectance from 10% to 12% after one hour exposure and an additional 4 to 9% after the second hour. After eighteen hours exposure all three coatings increased in reflectance from 6% to 8% over the second hour's exposure and, at the end of one hundred hours exposure there was an additional 4 to 5% increase in reflectance for the Z-6020 and the A-1100. The XA-1902 catalyzed coating upon removal from the furnace completely flaked off in large ribbons. It appears that the XA-1902 catalyst is unsatisfactory for the temperature involved. Because of the severe drop in reflectance after one hour exposure, it was thought that the discoloration could be due to the catalyst. Acting on this assumption smear samples of each of the three catalysts were exposed to temperature for one hour for color comparison. The Z-6020 catalyst turned a very dark brown, almost black. The A-1100 catalyst turned off-white to a later dark brown, while the XA-1902 changed to a light tan which did not change with additional exposure.

In order to have more than one manufacturer and one resin available, a series of resins manufactured by General Electric were evaluated to determine if their silicone resins could be catalyzed with the A-1100 amine. The General Electric resins included SR-82, SR-111, SR-112, SR-119, SR-120 and SR-223. These coatings were catalyzed at 5, 10, 15, 20 and 25 percent with A-1100 amine catalyst as clear coatings.

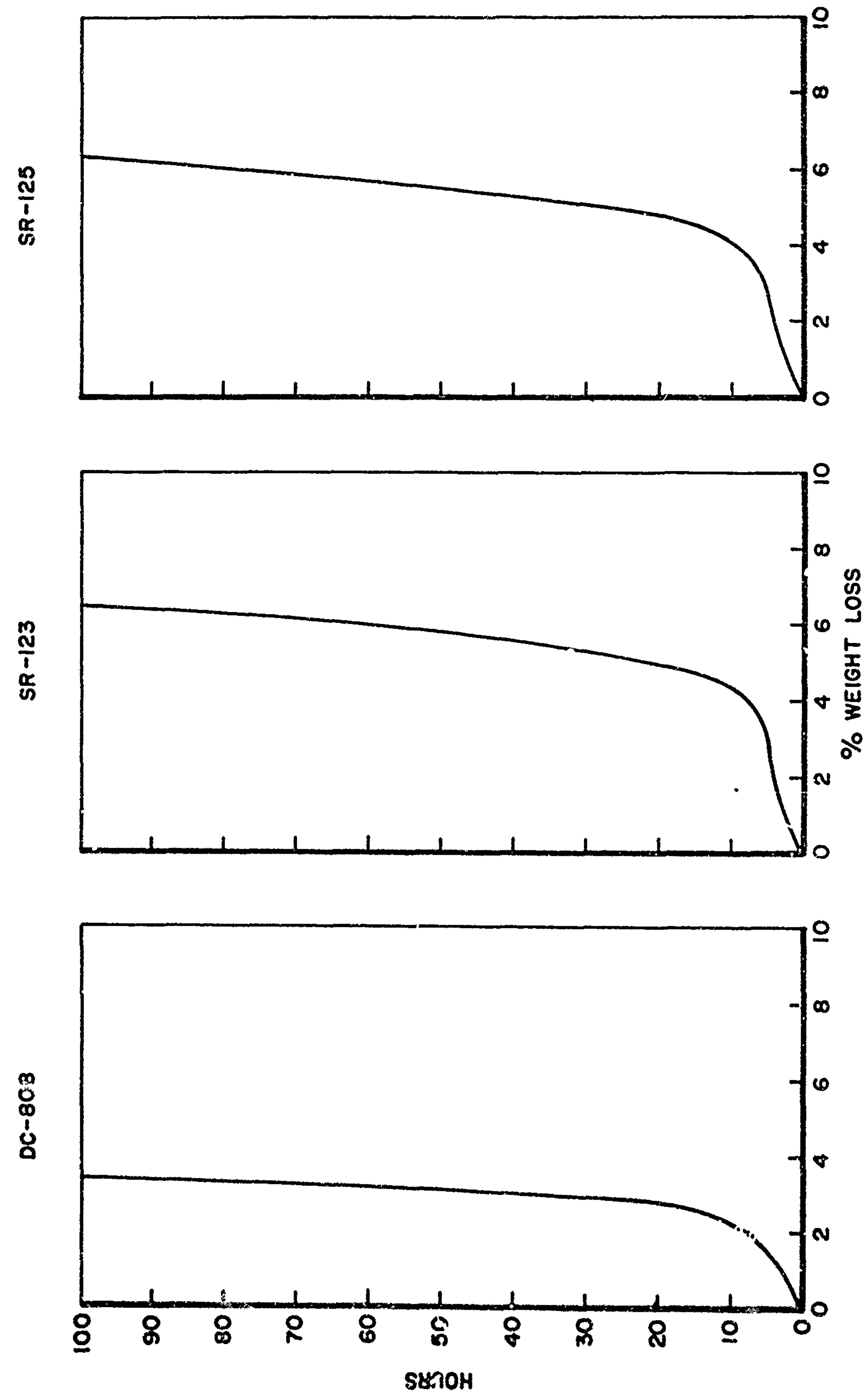

FIGURE 12 - The Percent Weight Loss of Clear Resins DC-808, SR-123 and SR-125 After $500^{\circ}F$ for 100 Hours.

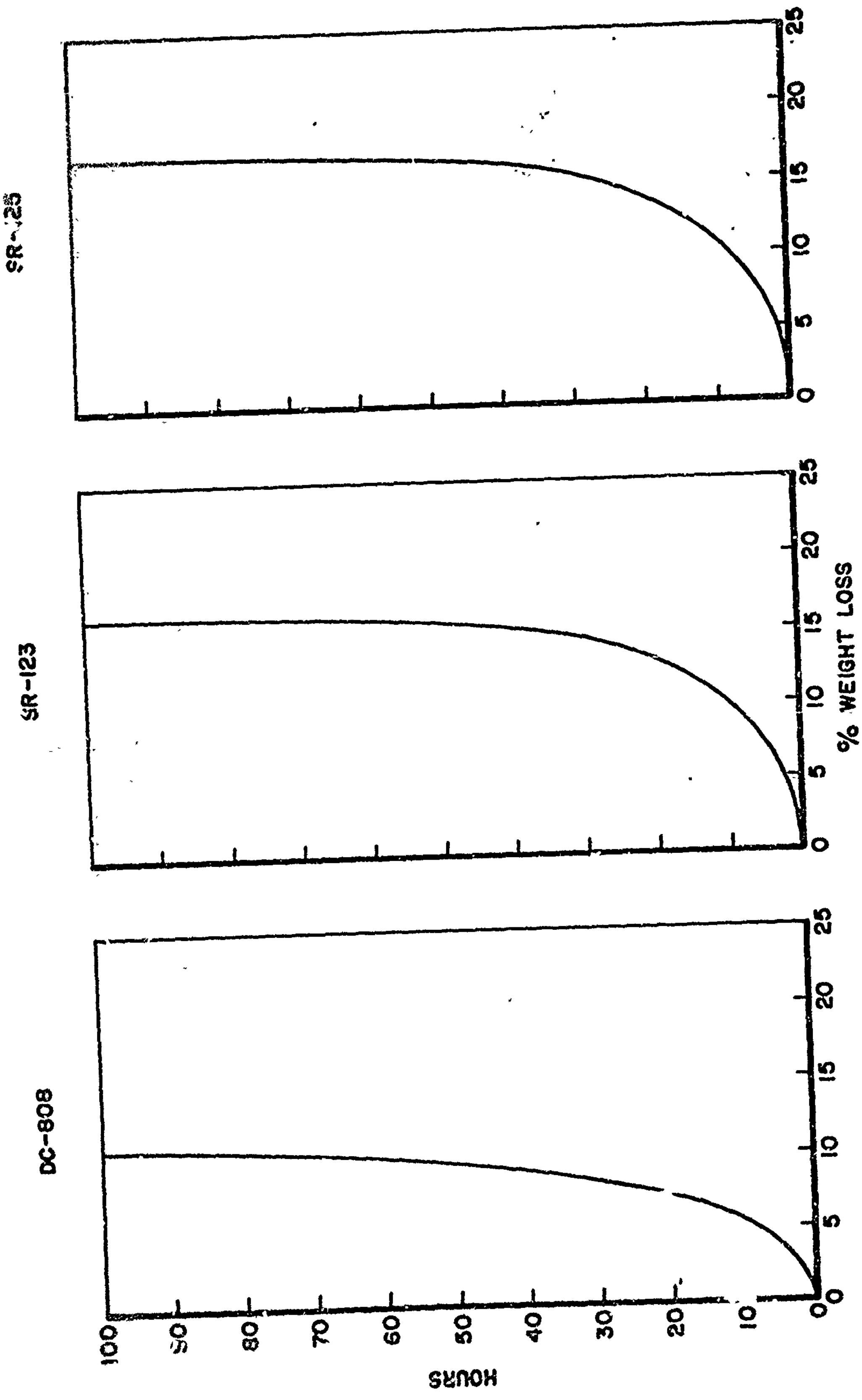

FIGURE 13 - The Percent Weight Loss of Clear Resins DC-808, SR-123 and SR-125 After 600°F for 100 hours.

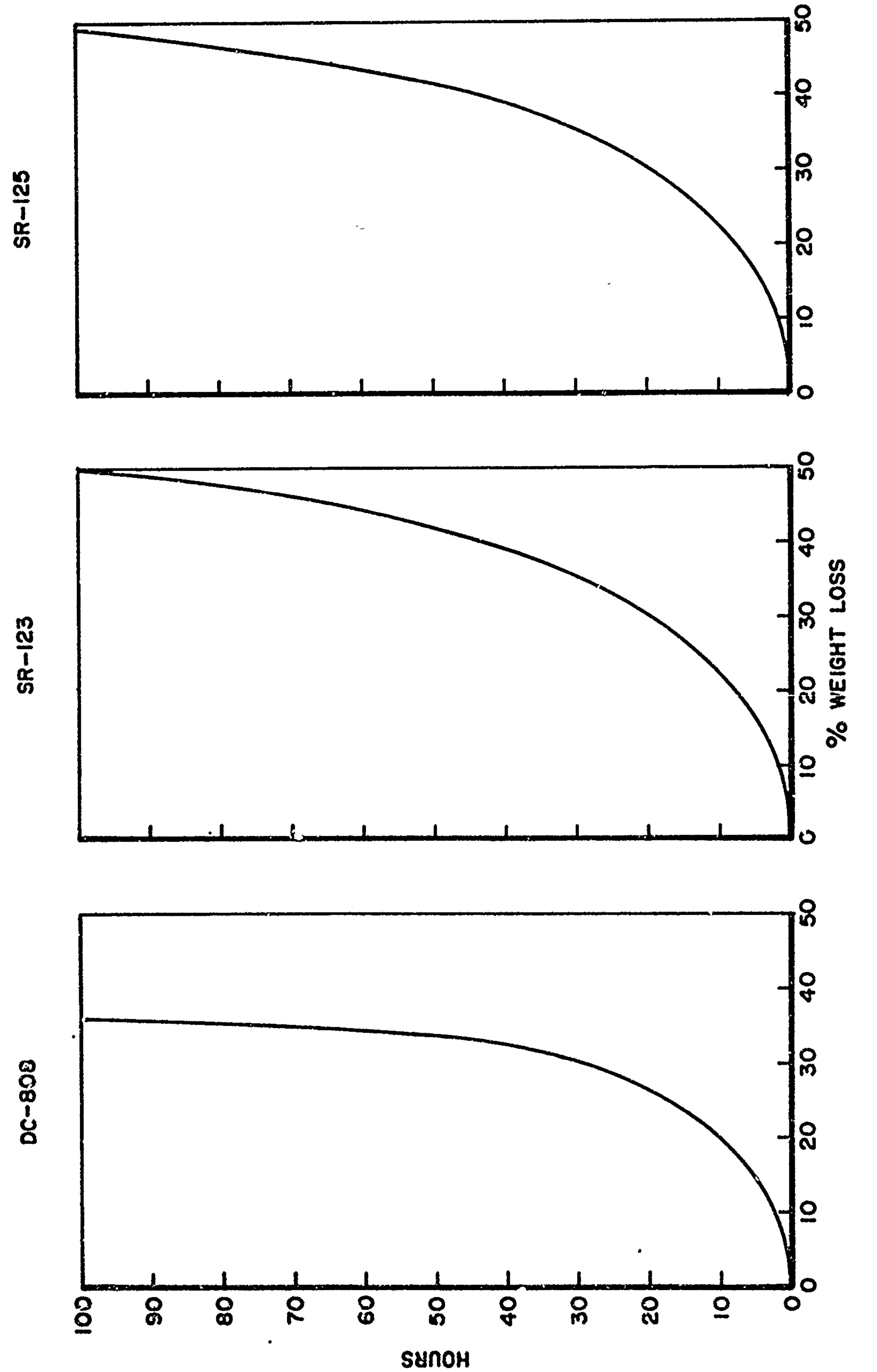

FIGURE 14 - The Percent Weight Loss of Clear Resins DC-808, SR-123 and SR-125 After 700°F for 100 hours.

Results were as follows:

1. Resin SR-82 catalyzed at 25% never fully cured.

2. Resin SR-111 catalyzed at 10% cured.

3. Resin SR-112 catalyzed at 25% cured but soft.

4. Resin SR-119 catalyzed at 10% cured.

5. Resin SR-120 catalyzed at 25% cured by soft.

6. Resin SR-223 catalyzed at 10% cured.

Samples of SR-111, SR-112, SR-119 and SR-223 were pigmented at a P/B ratio of 100/100 with R-900 titanium dioxide. Upon exposure to 650°F, for two hours, the SR-111, SR-112 and SR-119 cracked severely and ribboned off of the panel. The SR-119 resin did withstand 650°F temperature exposure for twenty-four hours; however, the coating turned a very pale tan with the initial reflectance of 91 percent dropping to 81 percent after two hours exposure and with an additional decrease in reflectance to 78 percent after twenty-four hours. The SR-223 withstood the 650°F temperature exposure for twenty-four hours. The initial reflectance of 94 percent dropped to 78 percent after exposure.

In order to have sufficient resin available for the preparation of Florida exposure panels a five gallon sample of SR-223 was ordered from General Electric only to be notified that SR-223 was no longer available but had been replaced with SR-123. This resin (SR-123) is exactly the same resin (on the solids basis) as was formerly supplied as SR-223. The only differenco is that SR-123 is a xylol solution while SR-223 was cut in toluol.

Since this research was conducted, General Electric has recently changed SR-123 to SR-125. The only change is that SR-125 is now supplied at 50% solids. In order to verify the high temperature stability of these new resins, weight losses were conducted at 500, 600 and 700°F for SR-123, SR-125 and DC-808. As can be seen in Figures 12, 13 and 14 the two General Electric resins are equal in temperature stability, although somewhat higher in weight loss at temperature than the Dow Corning DC-808.

Formulations No. AF-66, AF-67 and AF-68-5 consisted of General Electric SR-123 resin pigmented at a P/B ratio of 25/100, 50/100 and 100/100 with Ti pure R-900 titanium dioxide.

G. CONCLUSIONS FROM RESIN STUDY

1. Of all the vehicles evaluated, Dow Corning DC-808 and General Electric SR-123 silicone resins are outstanding for their thermal resistance properties and will produce coatings that can be room temperature cured using an amine silane catalyst.

2. Two amino-functional materials consisting of Z-6020 and A-1100 will, when properly compounded, provide an air dry room temperature cured unmodified silicone coating.

3. Using Dow Corning DC-808 and General Electric SR-123 resins, the following formulations will provide reflectances exceeding 85 percent after one hundred hours heat exposure at 460°F:

AF-47
AF-58
AF-58-5
AF-66
AF-67
AF-68-5

SECTION IV

PRIMER INVESTIGATIONS

A. INTRODUCTION

Corrosion is the chemical interaction of a metal or alloy with its environment. For present day aircraft, the environment can be dry and sunny and a few flight hours later can find the aircraft in a salt water atmosphere. Because of these various environmental conditions, most of the metal components are painted with a corrosion inhibiting primer and several top coats to provide a barrier coat against moisture and ionic penetration. The well known and excellent zinc chromate pigment has for many years, when properly applied as a primer coating, provided satisfactory performance. However, with the increased speeds at which aircraft are now operating and the increased skin temperatures due to aerodynamic heating, the limitation of the zinc chromate pigments have been surpassed. Based on information obtained from a literature survey, the best of the corrosion inhibitive primers for steel contain approximately forty to fifty-five percent of the basic inhibitive pigments while the remaining percentages serve as fillers and extenders. With this information together with past experience gained on the unmodified silicone resins, the only logical starting point in obtaining a balanced paint system would be using the same silicone resins for the primer vehicle.

B. EXPERIMENTAL FORMULATIONS

Using the DC-808 unmodified silicone resin and catalyzed with Z-6020 at three percent based on the resin solids, primer formulations AF-P1, AF-P2 and AF-P3 were compounded using standard iron oxide and zinc chromate as the inhibitive pigments with magnesium silicate as the extender holding the P/B ratio at 100/100. Primer No. AF-P1 resulted in a light pink colored coating which after heat exposure for twenty-four hours at 650°F produced a very dark red, very rough surfaced coating. Primer No. AF-P2 which had five percent less magnesium silicate gave a little better grind with the same color. Heat resistance of this coating was the same as Primer No. AF-P1. Primer AF-P3 had twelve percent less magnesium silicate than AF-P2. Results were more favorable in grind, heat stability and surface wetting.

Formulations No. AF-P4 and AF-P5 were compounded with essentially the same pigments as were AF-P1, AF-P2 and AF-P3 except that AF-P4 had twenty percent titanium dioxide and AF-P5 had thirty-six percent titanium dioxide to increase the reflectance. Although the color was slightly improved it is doubtful if these small percentages of titanium dioxide will affect the overall performance after a top coat has been applied.

Based on information received from the Mineral Pigments Corporation, three relatively new corrosion inhibitive pigments, specifically calcium, strontium and zinc molybdates, were compounded into three primer formulations using these pigments in the pure state rather than as the extended materials to determine their color stability. For applications up to 600°F each of these would be a satisfactory inhibitor in terms of thermal stability and protection against corrosion. A P/B ratio of 200/100 was maintained for all three formulations. This ratio resulted in the calcium and strontium formulations being a semi-gloss whereas the zinc molybdate gave a rather high gloss. The reflectance results were as indicated in Table VIII:

TABLE VIII

Percent Reflectance After Heat Exposure at 650°F

Formula Number	Original	1 Hour	3 Hours	24 Hours
P-6-Z	90	75	75	78
P-6-C	89	70	70	Severe flaking
P-6-S	89	72	72	Severe flaking

Based on the above results a series of ten primer formulations were compounded using DC-808 and G. E. SR-223 silicone resins for evaluation in the salt fog cabinet. Two P/B ratios of 100/100 and 150/100 were maintained throughout this evaluation. The inhibitive pigments were red lead and zinc molybdate extended on calcium carbonate with various percentages of zinc chromate, zinc oxide, magnesium silicate and diatomaceous silica for fillers.

The Z-6020 amine catalyst was not used for this series of primer formulations because it was found that the A-1100 amine catalyst, although it required a higher percentage to accomplish the same drying time, resulted in a much whiter coating at elevated temperatures. In all cases the catalyst used was A-1100 at ten percent based on resin solids. In order to determine the effectiveness of the inhibitive properties of the primer, common 10-10 cold rolled steel (well known for its rapid corrosion properties) was used as the substrate. All formulations were sprayed to a dry film thickness of 1.0 ± 0.2 mil and permitted to air dry for ten days prior to evaluation. The scribe mark and operation of the twenty percent salt fog cabinet was in accordance with Federal Test Method Standard TT-P-141a, Method 6061.

C. RESULTS

Formulations AF-P6 and AF-P7 (DC-808 and G. E. SR-223) failed the tape test at the end of twenty-four hours. Since there was no evidence of corrosion the panels were returned for an additional twenty-four hours. After removal, the panels were examined and it was noted that there were small scattered blisters covering the entire panel. No further evaluation of these formulations were conducted. At the end of seventy-two hours, Formulation No. AF-P13 had large scattered broken blisters.

Formulations AF-P8 through AF-P12 were satisfactory for seventy-two hours but at the end of ninety-six hours the coatings were rather soft. There were several small blisters very near and slight rusting at the scribe mark of Formulations AF-P8, AF-P9 and AF-P10. Because of the location of the blisters, the panels were returned for continued exposure. There were no changes noted until at the end of two hundred and fifty hours at which time the following changes were noted:

Formulation	Remarks
AF-P8	Small blisters at scribe
AF-P9	Original blisters disappeared
AF-P10	Original blisters disappeared
AF-P11	Medium scattered blisters
AF-P12	Scattered blisters.

Twenty-four hours after removal the coatings regained their original hardness.

Formulations AF-P14 (SR-223) and AF-P15 (DC-808) pigmented at a P/B ratio of 100/100 using 64% zinc molybdate, 36% magnesium silicate and diatomaceous silica (1:1), at the end of twenty-four hours showed initial rusting at the scribe mark, but otherwise they were in excellent condition. Additional exposure to two hundred hours failed to produce any additional failures. Due to the immediate need for a primer and top coat to be applied on a test aircraft, and the considerable amount of time involved in the preparation of exposure panels for Florida weathering, no further primer formulations were evaluated.

SECTION V

FLORIDA WEATHERING

A. BACKGROUND

In order to determine the usefulness of any experimental coating before the preparation of a specification and subsequent release to a using activity, the coating must demonstrate its ability to provide satisfactory performance under a variety of adverse climatic conditions. The most severe conditions a coating can be subjected to is twelve to twenty-four months weathering in a marine atmosphere. This type of environment consisting of high temperature and humidity, rain, high solar radiation, moist salt atmosphere, and cooling at night produces the most severe service conditions any coating is likely to be subjected to in actual service. Furthermore, these climatic conditions cannot be duplicated within the laboratory to any satisfactory degree. All experimental panels referenced in this section were exposed twenty-four months at the Naval Bureau of Weapons exposure site located on Fisher Island approximately three hundred yards south of Miami Beach, Florida.

Inspection and evaluation of all panels was conducted quarterly for the first year's exposure, and semi-annually for the second year's exposure for the following properties:

1. Reflectance*
2. Gloss 60° *
3. Adhesion Dry Tape Test
4. Corrosion
5. Dirt Retention
6. Blistering
7. Color Change
8. Chalking
9. Cracking

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*Gardner Portable Glossmeter and Reflectometer

10. Peeling

11. Fungus Growth

B. SUBSTRATES

The substrates selected for this evaluation were recommended by the Materials Application Division (MAAS) as metals which have potential use as skin coverings for high speed aircraft. In addition, one substrate, 2024 clad aluminum, was included for reference purposes. All panels were cut 5" x 16" x 0.032-0.040". The actual thickness was dependent on the material available. The substrates were:

1. 13V 11Cr 3Al Titanium
2. 6Al 4V Titanium
3. 301 Stainless Steel
4. 2024 Clad Aluminum Alloy

C. SUBSTRATE PREPARATION

All evaluation panels prior to coating received the following treatment:

1. Solvent cleaned.
2. Vapor degreased.
3. Two minute washing in alcoholic phosphoric acid prepared in accordance with Federal Test Method Standard No. 141a, 2013.1, Table II.
4. Tap water wash.
5. Distilled water rinse.
6. Water break check.
7. Excess water blown off with compressed air.
8. Oven dried at 212° F

9. Stored in dust free cabinet until sprayed.

10. All clad aluminum alloy panels after step No. 6 received a three-minute immersion in Iridite 14* conforming to Specification MIL-C-5541. Steps No. 4 thru 9 were then completed.

D. APPLICATION OF COATINGS

1. All coatings were applied by spray application.

2. The silicone pigmented primers were catalyzed with the A-1100 amine catalyst at a ratio of ten percent based on resin solids. The catalyzed mixture was allowed to stand for one hour before reducing the viscosity for spray application to twenty-six seconds on a No. 4 Ford cup. The primer coatings were permitted to air dry for eighteen hours before application of the topcoats. All silicone topcoats were catalyzed as were the primer coats using the same catalyst and percentage. The white topcoats were applied to a total dry film thickness of 2.5 ± 0.3 mils.

E. MASTER CODE SHEET

All information related to the exposure panels regarding the various substrates, primer and topcoat formulations can be found in Appendix I.

F. EXPOSURE DATA

All information relating to the exposure data for all formulations from May 1965 to May 1967 can be found in Appendix II. For simplification in evaluation, all exposure panels having the same topcoat formulations have been grouped together. A consolidation of the exposure data for all formulations is as follows:

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*Manufactured by Allied Research Products, Inc.

TABLE IX

Consolidation of Florida Exposure Data

	REFLECTANCE		60° GLOSS	
Formula	Original	24 Months	Original	24 Months
AF-47	86	88	91	82
AF-58	86	87	85	78
AF-58-5	85	87	85	85
AF-66	87	89	95	74
AF-67	90	90	95	88
AF-68-5	89	90	91	77

G. CONCLUSIONS

Based on the twenty-four months exposure data contained herein the following conclusions are made:

1. All primer and topcoat formulations exhibited excellent weathering ability.

2. Formulation No. AF-58-5 was the only topcoat formulation which maintained its original gloss.

3. Formulation No. AF-67 maintained its original reflectance but decreased 7 percent in gloss. This slight decrease was not severe in light of its initial higher gloss.

4. Formulation No. AF-66 increased its reflectance by two percent but had the highest(21%) loss in gloss.

5. All topcoat formulations maintained or for unknown reasons increased their reflectance.

6. All stainless steel exposure panels showed considerable edge and back rusting. The rust stain which ran down onto the coatings was easily removed with an abrasive (Ajax) cleaner.

7. The backsides of all 2024 clad aluminum alloy panels were severely pitted.

8. All titanium panels were in very good condition.

9. The adhesion of all coatings was excellent with no failures.

SECTION VI

FINAL LABORATORY EVALUATION

A. INTRODUCTION

The original laboratory evaluation of all formulations was not intended to be so extensive. It was anticipated that after two years weathering in a Florida marine atmosphere that several of the formulations and many exposure panels would for various reasons result in failures. However, after two years of Florida weathering none of the original ninety-six panels had any form of failure other than a slight loss in sixty degree gloss.

Therefore, due to the lack of failure during exposure it was necessary to prepare duplicate panels for laboratory evaluation. Since all of the formulations were satisfactory in the salt spray, and adhesion screening, the remaining criteria was the elevated temperature determination. All panels were prepared in accordance with the master code sheet for Florida weathering (See Appendix I).

B. METHOD OF HIGH TEMPERATURE EVALUATIONS

All panels exposed to elevated temperatures were subjected to the following temperature cycle:

1. Fifty hours at 500°F

2. Panels removed, examined and measured for reflectance and gloss.

3. Returned for additional fifty hours at 500°F.

4. The above procedure was followed for all temperature exposures through 700°F, increasing the temperature every one hundred hours in increments of fifty degrees.

If at any time during the high temperature evaluation any failures were observed, the panel was removed and the failure noted. Due to the extreme temperature (700°F) to which these coatings were subjected all coatings failed for one or more reasons. The type of failure and temperature at which the failure occurred can be found in Appendix III.

In order to determine the long term stability of the various formulations, four representative panels of each formulation were subjected to 460°F for 1000 hours. Reflectance and 60° gloss measurements were taken at 250, 500, 750 and 1000 hours exposure. Results of this evaluation can be found in Appendix IV.

C. METHOD OF EVALUATING FUEL RESISTANCE

1. In order to determine the synthetic lubricant resistance of the various formulations, three different test fluids were used for this evaluation. They consisted of Specification MIL-L-7808 Synthetic Lubricant, Hydrocarbon Test Fluid Type II and Specification TT-T-656 Tricresyl Phosphate. All panels were immersed to one-half of their length in each fluid for twenty-four hours. All panels were examined immediately after removal from the fluid for peeling, yellowing, blistering and other film defects. Seventy-two hours after removal the panels were evaluated for pencil hardness and other film defects. Results of this evaluation can be found in Appendix V.

2. The following table lists the pencil hardness scale that was used during this evaluation:

TABLE X

Pencil Hardness Scale

2H	Hard	2B	Soft +
H	Medium	3B	Very Soft
F	Intermediate	4B	Extra Soft
HB	Medium Soft	5B	Extremely Soft
B	Soft	6B	Softest

3. In order to determine the effect of post curing at elevated temperature after immersion, Formulations No. AF-47 and AF-66 were subjected to 460°F for thirty minutes, then examined for pencil hardness. Results of this evaluation can be found in Appendix V.

D. ACCELERATED WEATHERING

Accelerated weathering or artificial weathering is the exposure of materials to intense ultraviolet light from closed carbon arc systems. Simulated rain and sunshine can be programmed in any manner desired. However, for this particular evaluation, a twin-arc Model DMC, weather-ometer was utilized with sunlight only. Results of this evaluation can be found in Appendix VI.

E. METHOD OF SALT SPRAY FOG EVALUATION

In this evaluation, all tests were conducted in accordance with Federal Test Method No. 141a, Method 6061, using a 5 percent salt fog.

F. METHOD USED TO DETERMINE EMITTANCE

The reflectance measurements from 1 to 15 microns were made on a Perkin-Elmer Model 13 Spectrophotometer using polished aluminum as a standard. The initial integrated spectral normal emittance was determined by calculations from the reflectance data. The spectral distribution of the energy radiated from 150°F body is given in Table XII. The spectral emittance was determined by subtracting the reflectance values from unity. The integrated total emittance was determined by dividing the curve into segments and determining the percent of energy reradiated at 150°F in each segment (as a percent of the total reradiated compared to a black body at the same temperature). These results were then summed up for each curve to obtain an integrated spectral emittance. For total emittance beyond 15 microns, an approximation had to be made. It can be seen from Table XII that 35.2 percent of the energy at 150°F is beyond 15 microns. A value of 0.23 was obtained by extrapolating the reflectance curve beyond 15 microns.

TABLE XI

Calculation of Normal Emittance at 80°F (27°C)

Wavelength Interval (μ)	% Normal Energy Emitted by Blackbody at 80°F
<5	1.4
5-6	2.8
6-7	4.4
7-8	5.8
8-9	6.6
9-10	7.0
10-11	5.5
11-12	6.5
12-13	6.0
13-14	5.0
14-15	5.0
>15	44.0

TABLE XII

Calculation of Normal Emittance at 150°F (66°C)

Wavelength Interval (μ)	% Normal Energy Emitted by Blackbody at 150°F
<4	0.6
4-5	2.3
5-6	4.5
6-7	6.3
7-8	7.5
8-9	7.5
9-10	7.6
10-11	7.0
11-12	6.6
12-13	5.2
13-15	9.7
>15	35.2

G. RESULTS OF HIGH TEMPERATURE EVALUATION

1. Exposure Temperature - 500°F

a. After 100 hours at temperature all formulations had a slight decrease in reflectance which varied from 5-7 percent for the DC-808 resin to 8-12 percent for the SR-123 resin.

b. The loss in 60° gloss was greater for the SR-123 resin which varied from 2-10 units than was the 2-6 unit loss for the DC-808 resin.

c. No failures were noted.

2. Exposure Temperature - 550°F

a. After 50 hours at temperature only four of the sixteen panels coated with Formulations No. AF-47 and AF-58-5 had not failed. The remaining panels failed after an additional 50 hours at temperature.

b. The remaining formulations all had a 1-2 percent increase in reflectance.

c. The loss in 60° gloss varied from 1-5 units.

3. Exposure Temperature - 600°F

a. Fifty hours exposure at temperature failed all but eight panels of Formulation AF-58. The remaining eight failed after an additional fifty hours at temperature.

b. One panel of Formulation AF-67 failed after fifty hours at temperature.

c. All formulations containing the DC-808 resin have now failed.

d. The reflectance remained essentially the same but there was a slight decrease in 60° gloss for the remaining formulations.

4. Exposure Temperature - 650°F

a. Fifty hours exposure failed all but six panels of Formulation AF-68-5. The remaining six failed after an additional fifty hours at temperature.

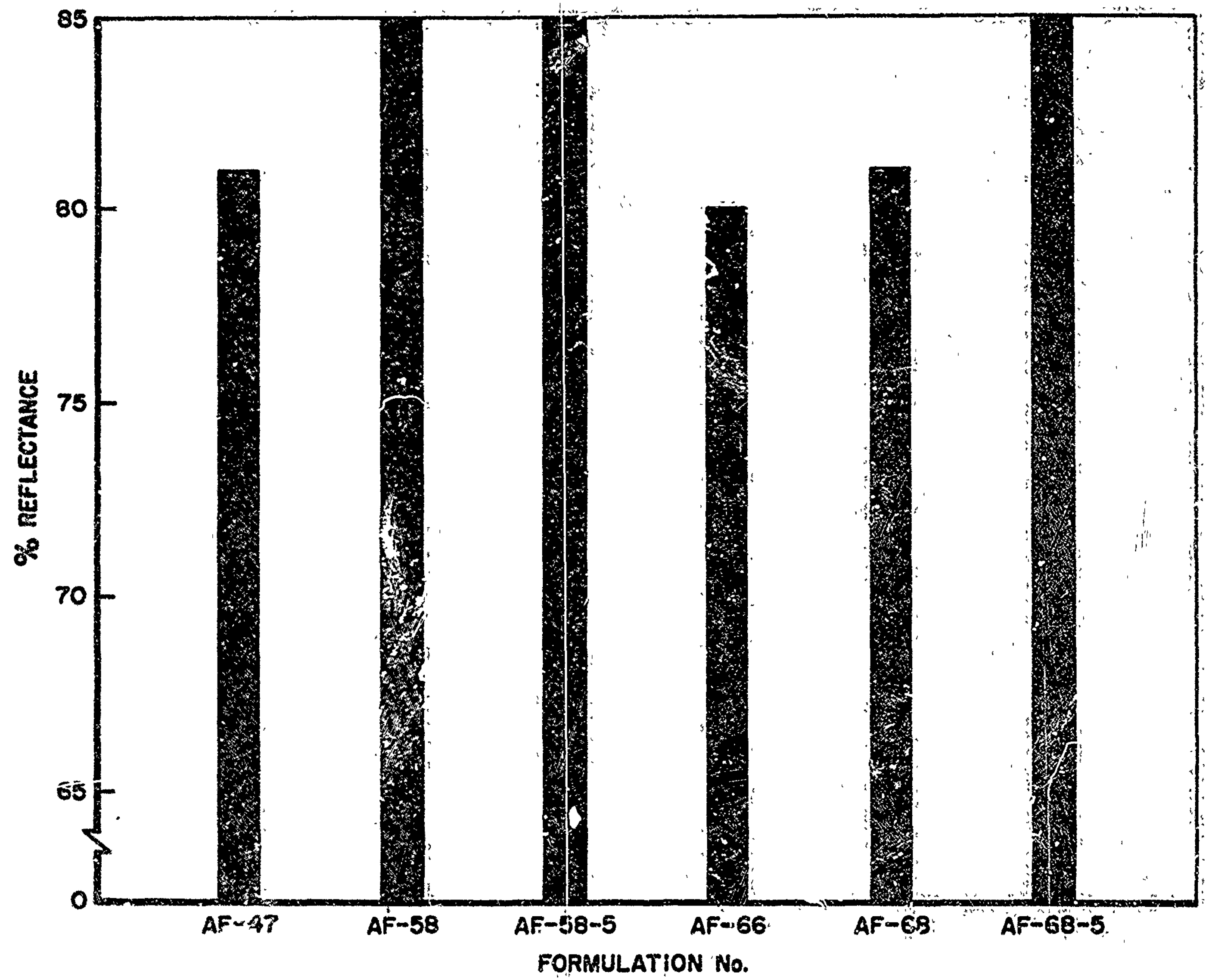

FIGURE 15 - Percent Reflectance After 1000 Hours at 460°F for Formulations AF-47, AF-58, AF-58-5 AF-66, AF-68 and AF-68-5

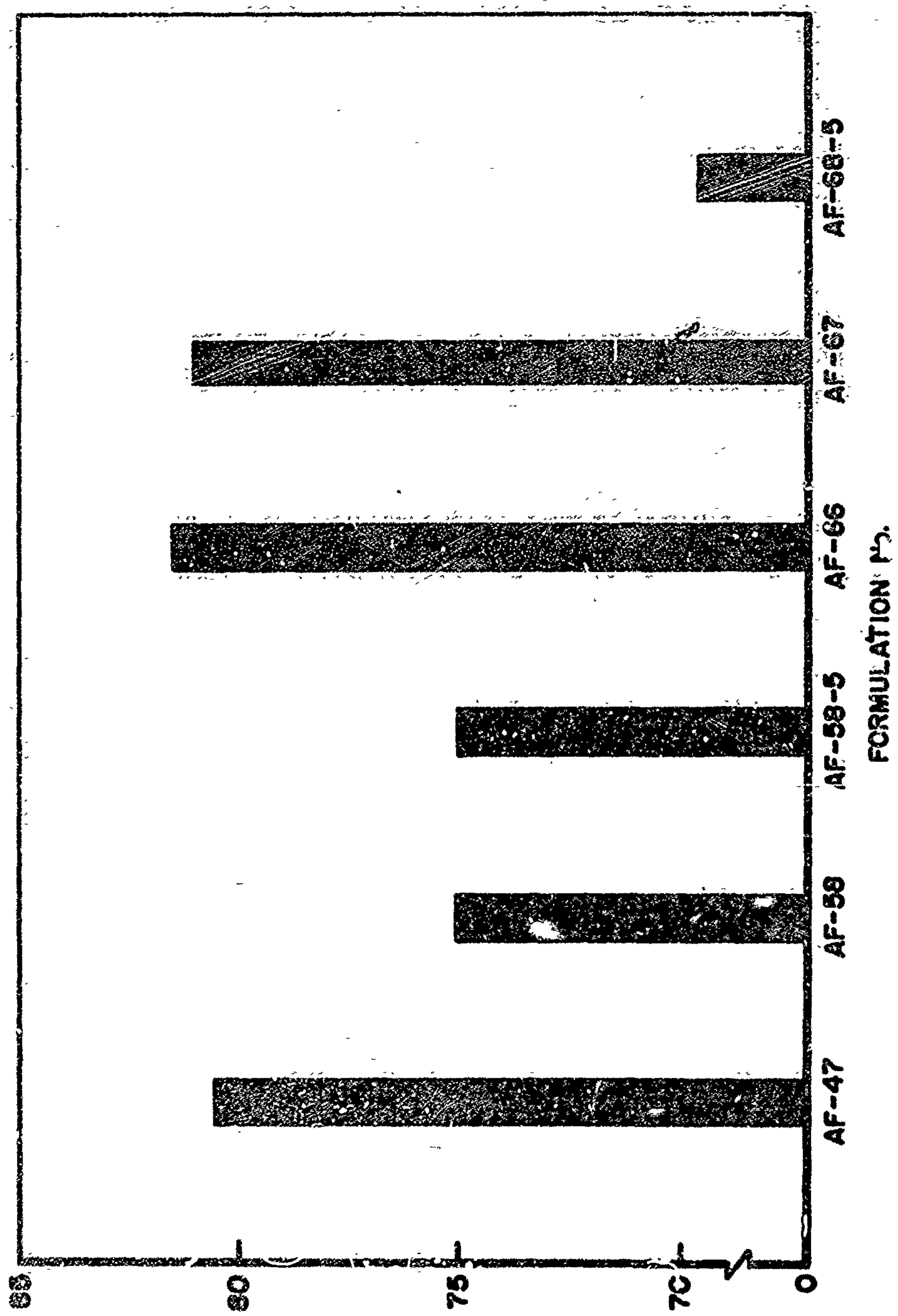

FIGURE 16 - 60° Gloss Reading After 1000 Hours at 460°F

b. Formulation AF-66 and AF-67 were satisfactory in all respects.

c. Both formulations had a slight increase in reflectance and a decrease in 60° gloss.

5. Exposure Temperature - 700°F

a. After fifty hours exposure all but five panels of Formulation No. AF-66 had failed. The remaining five failed with an additional fifty hours exposure.

b. All the panels coated with Formulation AF-67 failed after fifty hours exposure.

H. RESULTS OF TEMPERATURE EXPOSURE AFTER 1000 HOURS AT 460°F

1. 60° Gloss

a. Formulation No. AF-47, AF-66 and AF-67 all had gloss ratings exceeding 80 units and would be satisfactory for field service.

b. Formulations AF-58, AF-58-5 and AF-68-5 varied from 69 to 75 units.

2. Reflectance

a. All six formulations had reflectance exceeding 79% and as high as 85%.

b. In summary, as can be seen in Figures 15 and 16, the lower P/B ratio coatings will provide higher gloss and lower reflectance whereas the higher the P/B ratio the reflectance will be greater and the gloss lower.

I. RESULTS OF THE FUEL RESISTANCE EVALUATION

1. Other than softening, no other film defects were noted on all panels exposed to specification MIL-L-7808 Synthetic Lubricant.

2. When subjected to Hydrocarbon Test, Fluid Type II, all panels coated with Formulation No. AF-58-5 had severe yellowing in addition to a slight softening.

3. Five panels consisting of Formulations No. AF-47, AF-58 and AF-58-5 all had small scattered blisters after removal from the hydrocarbon test fluid. Since these were the only panels which had any form of blistering of the ninety-six evaluated, there is no logical explanation for these failures.

4. Other than slight softening, no film defects were noted when subjected to Specification TT-T-656 Tricresylphosphate.

5. Post curing of Formulations No. AF-47 and AF-66 after immersion revealed that the original pencil hardness was regained for all fuels except the tricresylphosphate which had a decrease of two pencil hardnesses for AF-66.

6. Those formulations which contained SR-123 resin were more susceptible to softening than were those containing the DC-808 resin when immersed in synthetic lubricant and tricresylphosphate.

7. Except for softening, all formulations except AF-58-5 would be satisfactory for field service.

J. REFLECTANCE AND 60° GLOSS AFTER 500 HOURS EXPOSURE IN THE WEATHEROMETER

The original plan was to expose the panels for one-thousand hours; however, after five-hundred hours there was such an insignificant loss of reflectance and gloss that all exposures were terminated at the end of five hundred hours.

K. RESULTS OF SALT SPRAY FOG EVALUATION

Duplicate heat exposure panels were prepared and subjected to a 5 percent salt fog exposure for 1000 hours. Of the panels exposed the only failures noted were on the aluminum substrate after 280 hours. The failure was not due to corrosion at the scribe line but due to blistering. The initial blisters were small but increased in size after continued exposure. The remaining panels exhibited no evidence of failures at the end of 1000 hours exposure.

L. RESULTS OF ACCELERATED WEATHERING

After 500 hours exposure in the Weatherometer, none of the coatings had yellowed. All panels exhibited less than 2 percent decrease in reflectance and only a 6 unit decrease in 60° gloss.

M. RESULTS OF EMITTANCE MEASUREMENTS

The calculated spectral emittance at $80^{\circ}F$ and $150^{\circ}F$ for formulation No. AF-66 which was representative for all white formulations evaluated under this program was 80 and 81 percent respectively.

N. CONCLUSIONS

Based on the laboratory data contained herein, the following conclusions are made:

1. Formulation No. AF-66 will withstand fifty hours exposure at $700^{\circ}F$. At temperatures exposures of $600^{\circ}F$ or lower this coating should perform indefinitely.

2. The reflectance of Formulation No. AF-66 after 100 hours exposure to $650^{\circ}F$ was 82 percent with a 60° gloss exceeding 80 units.

3. After temperature exposure for 1000 hours at $460^{\circ}F$ it was found that the higher the pigment loading the higher the reflectance. However the reverse was true for the 60° gloss readings.

4. All formulations evaluated for 1000 hours at $460^{\circ}F$ were satisfactory. The reflectances varied from 80-85 percent and the 60° gloss readings were between 69 and 83 units.

5. Results of the fuel resistance evaluation indicate that formulations which contained SR-123 resin were more susceptible to softening than were those containing the D -808 resin when immersed in synthetic lubricant and tricresylphosphate.

6. Except for softening, all formulations except AF-58-5 (which discolored in Type II fluid) would be satisfactory for field service where the maximum temperature would not exceed $500^{\circ}F$.

7. Formulations No. AF-47 and AF-66 (representatives of both resins) regained their original pencil hardness after thirty minutes exposure at $460^{\circ}F$ following fuel immersion.

8. All coatings were satisfactory after 500 hours exposure to accelerated weathering.

9. All coatings applied to aluminum substrates failed by blistering after 280 hours exposure to a 5% salt fog.

10. All coatings applied to 13V 11Cr 3Al Titanium, 6Al 4V Titanium and 301 stainless steel substrates were satisfactory after 1000 hours salt fog exposure.

SECTION VII

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APPENDIX I

Master Code Sheet

APPENDIX I

MASTER CODE SHEET

Panel No.	Substrate	Primer	Top Coat Resin	Formula No.
1	1	AF-P14	GE-123	AF-66
2	1	"	"	-67
3	1	"	"	-68-5
4	1	AF-P15	"	-66
5	1	"	"	-67
6	1	"	"	-68-5
7	1	AF-P9	"	-66
8	1	"	"	-67
9	1	"	"	-68-5
10	1	AF-P10	"	-66
11	1	"	"	-67
12	1	"	"	-68-5
13	1	AF-P14	DC-808	-47
14	1	"	"	-58
15	1	"	"	-58-5
16	1	AF-P15	"	-47
17	1	"	"	-58
18	1	"	"	-58-5
19	1	AF-P9	"	-47
20	1	"	"	-58
21	1	"	"	-58-5
22	1	AF-P10	"	-47
23	1	"	"	-58
24	1	"	"	-58-5
25	2	AF-P14	GE-123	-66
26	2	"	"	-67
27	2	"	"	-68-5
28	2	AF-P15	"	-66
29	2	"	"	-67
30	2	"	"	-68-5
31	2	AF-P9	"	-66
32	2	"	"	-67
33	2	"	"	-68-5
34	2	AF-P10	"	-66
35	2	"	"	-67
36	2	"	"	-68-5
37	2	AF-P14	DC-808	-47
38	2	"	"	-58
39	2	"	"	-58-5
40	2	AF-P15	"	-47

Panel No.	Substrate	Primer	Top Coat Resin	Formula No.
41	2	AF-P15	DC-808	AF-58
42	2	"	"	-58-5
43	2	AF-P9	"	-47
44	2	"	"	-58
45	2	"	"	-58-5
46	2	AF-P10	"	-47
47	2	"	"	-58
48	2	"	"	-58-5
49	3	AF-P14	GE-123	-66
50	3	"	"	-67
51	3	"	"	-68-5
52	3	AF-P15	"	-66
53	3	"	"	-67
54	3	"	"	-68-5
55	3	AF-P9	"	-66
56	3	"	"	-67
57	3	"	"	-68-5
58	3	AF-P10	"	-66
59	3	"	"	-67
60	3	"	"	-68-5
61	3	AF-P14	DC-808	-47
62	3	"	"	-58
63	3	"	"	-58-5
64	3	AF-P15	"	-47
65	3	"	"	-58
66	3	"	"	-58-5
67	3	AF-P9	"	-47
68	3	"	"	-58
69	3	"	"	-58-5
70	3	AF-P10	"	-47
71	3	"	"	-58
72	3	"	"	-58-5
73	4	AF-P14	GE-123	-66
74	4	"	"	-67
75	4	"	"	-68-5
76	4	AF-P15	"	-66
77	4	"	"	-67
78	4	"	"	-68-5
79	4	AF-P9	"	-66
80	4	"	"	-67
81	4	"	"	-68-5
82	4	AF-P10	"	-66
83	4	"	"	-67
84	4	"	"	-68-5
85	4	AF-P14	DC-808	-47

Panel No.	Substrate	Primer	Top Coat Resin	Formula No.
86	4	AF-P14	DC-808	AF-58
87	4	"	"	-58-5
88	4	AF-P15	"	-47
89	4	"	"	-58
90	4	"	"	-58-5
91	4	AF-P9	"	-47
92	4	"	"	-58
93	4	"	"	-58-5
94	4	AF-P10	"	-47
95	4	"	"	-58
96	4	"	"	-58-5

TABLE XIII

SUBSTRATE CODE

1. 13V11Cr3A1 Titanium
2. 6A14V Titanium
3. 301 Stainless steel
4. 2024 Clad Aluminum Alloy

TABLE XIV

PIGMENT/BINDER RATIO

Top Coat	
Formula No.	P/B
AF-66	25/100
AF-67	50/100
AF-68-5	100/100
AF-47	25/100
AF-58	50/100
AF-58-5	100/100
Primer	
AF-P9	150/100
AF-P10	100/100
AF-P14	100/100
AF-P15	100/100

APPENDIX II

Florida Weathering

TABLE XV
Florida Weathering of Formulation No. AF-47 after 24 Months Exposure

Panel No.	Reflectance Months			60° Gloss Months		
AF	Orig.	12	24	Orig.	12	24
13	85	85	88	91	80	83
16	85	84	88	91	82	83
19	85	84	89	90	82	83
22	85	84	88	90	82	82
37	87	85	88	92	81	82
40	85	83	88	92	82	82
43	87	84	88	91	81	82
46	87	84	88	91	82	82
61	85	84	89	92	82	82
64	86	83	88	91	80	83
67	85	84	88	90	81	83
70	85	83	88	91	82	83
85	86	83	88	92	82	82
88	87	84	88	91	82	82
91	87	83	88	90	82	82
94	86	83	68	90	82	82

TABLE XVI
Florida Weathering of Formulation No. AF-58 after 24 Months Exposure

Panel No.	Reflectance Months			60° Gloss Months		
AF	Orig.	12	24	Orig.	12	24
14	87	85	87	85	75	78
17	84	82	86	84	75	78
20	84	84	86	85	75	78
23	85	84	87	84	75	78
38	86	84	87	86	75	78
41	88	85	88	86	76	79
44	87	85	87	85	75	79
47	87	85	87	86	74	79
62	86	85	85	86	75	78
65	84	84	87	85	75	78
68	84	84	86	84	75	78
71	84	84	86	86	75	79
86	88	86	88	86	75	78
89	88	86	88	86	75	78
92	87	85	88	86	75	78
95	87	84	88	84	75	79

TABLE XVII

Florida Weathering of Formulation No. AF-58-5 after 24 months exposure

Panel No.	Reflectance Months			60° Gloss Months		
AF	Orig.	12	24	Orig.	12	24
15	85	84	88	84	82	85
18	84	84	87	86	82	85
21	84	84	88	85	81	84
24	85	84	87	84	81	85
39	87	85	87	86	82	85
42	87	84	87	86	81	85
45	87	84	87	86	80	84
48	84	82	86	84	80	84
63	86	85	87	86	81	85
66	85	84	89	86	82	85
69	84	84	88	84	82	85
72	84	82	88	84	80	84
87	87	85	88	86	82	85
90	87	85	87	84	82	85
93	87	84	88	85	82	85
96	87	85	87	84	80	84

TABLE XVIII

Florida Weathering of Formulation No. AF-66 after 24 months Exposure

Panel No.	Reflectance Months			60° Gloss Months		
AF	Orig.	12	24	Orig.	12	24
1	88	85	88	95	86	75
4	89	84	90	96	85	75
7	87	84	89	95	85	75
10	87	85	88	95	83	73
25	88	85	90	95	85	74
28	88	84	88	96	85	75
31	87	86	88	95	85	74
34	88	85	90	95	85	75
49	87	85	88	95	83	73
52	87	86	88	95	83	74
55	86	84	88	96	84	74
58	87	85	89	95	83	73
73	87	85	90	95	84	73
76	88	85	90	95	83	74
79	86	85	89	95	82	75
82	87	85	88	95	84	74

TABLE XIX

Florida Weathering of Formulation No. AF-67 after 24 Months Exposure

Panel No.	Reflectance Months			60° Gloss Months		
AF	Orig	12	24	Orig	12	24
2	90	85	90	95	85	89
5	89	85	90	95	85	88
8	90	85	90	95	85	89
11	90	85	89	95	85	89
26	90	85	89	94	85	88
29	90	85	89	95	85	88
32	90	86	89	95	85	89
35	90	86	90	95	85	88
50	90	85	90	95	85	88
53	89	85	89	95	85	89
56	89	84	90	95	85	89
59	89	84	90	95	84	88
74	89	85	90	95	85	89
77	89	85	90	95	85	88
80	89	85	89	95	85	87
83	89	85	89	95	85	88

TABLE XX

Florida Weathering of Formulation No. AF-68-5 after 24 Months Exposure

Panel No.	Reflectance Months			60° Gloss Months		
AF	Orig	12	24	Orig	12	24
3	87	85	89	91	80	77
6	87	86	89	90	81	77
9	89	85	89	91	82	76
12	89	86	90	90	80	77
27	87	86	89	91	81	77
30	86	85	89	90	81	76
33	90	87	90	90	80	76
36	89	87	90	91	80	76
51	89	88	89	91	81	75
54	91	88	89	91	80	76
57	89	88	90	91	80	77
60	89	88	90	91	80	77
75	89	86	90	90	80	77
78	89	86	90	92	81	77
81	91	86	89	91	81	76
84	91	86	90	91	81	77

APPENDIX III

High Temperature Evaluation

APPENDIX III

HIGH TEMPERATURE EVALUATION

TABLE XXI

Reflectance and 60° Gloss of Formula No. AF-47 After 100 Hours at 500°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60° Gloss Orig	60° Gloss 50 hrs.	60° Gloss 100 hrs.
13	86	78	80	88	87	88
16	87	78	80	87	87	87
19	87	79	81	88	87	87
22	86	78	80	89	88	87
3[illegible]	86	78	79	89	87	87
40	86	78	80	91	89	88
43	86	78	80	89	87	86
46	88	79	80	90	85	85
61	86	78	80	88	87	86
64	88	78	79	87	87	86
67	86	78	80	88	87	86
70	87	79	81	88	87	86
85	87	77	79	89	86	88
88	87	78	80	86	86	86
91	88	79	81	90	86	88
94	88	79	80	90	87	88

TABLE XXII

Reflectance and 60° Gloss of Formula No. AF-58 After 100 Hours at 500°F

Panel No	Reflectance Orig.	Reflectance 50 Hrs.	Reflectance 100 Hrs.	60° Gloss Orig.	60° Gloss 50 hrs.	60° Gloss 100 hrs.
14	90	81	82	85	81	82
17	90	81	83	82	80	80
20	90	81	82	82	81	81
23	90	81	82	83	81	81
38	91	81	82	84	81	81
41	90	81	83	83	81	80
44	91	81	83	85	82	81
47	91	80	83	84	81	80
62	91	81	82	85	82	82
65	90	81	83	85	82	81
68	90	81	83	85	82	81
71	89	81	83	85	81	80
86	90	80	82	85	80	81
89	89	81	82	83	82	82
92	90	82	83	84	80	81
95	91	81	83	87	8[illegible]	82

TABLE XXIII

Reflectance and 60° Gloss of Formula No. AF-58-5 after 100 hours at 500°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
15	90	84	85	87	82	82
18	90	83	84	87	83	82
21	89	84	85	86	80	80
24	87	85	86	88	81	82
39	91	84	85	87	82	82
42	90	84	85	87	82	81
45	91	84	86	89	83	82
48	91	84	85	87	82	80
63	91	84	85	87	82	80
66	91	84	85	88	84	82
69	92	85	86	88	84	82
72	91	85	86	87	84	82
87	90	84	85	87	81	81
90	90	84	85	87	80	80
93	90	84	85	87	80	80
96	91	84	85	89	83	83

TABLE XXIV

Reflectance and 60° Gloss of Formula No. AF-66 after 100 hours at 500°F

Panel No.	Reflectance			60° Gloss		
	Orig	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
1	88	73	75	91	[illegible]	90
4	89	73	75	93	[illegible]	90
7	87	74	76	91	91	90
10	88	73	75	92	91	90
25	88	74	75	90	91	90
28	87	74	76	93	90	90
31	87	75	76	93	89	90
34	89	75	76	91	91	90
49	88	74	75	88	89	88
52	88	74	76	92	90	89
55	88	75	77	89	89	89
58	88	74	77	92	91	90
73	86	73	75	92	92	91
76	87	73	74	92	91	91
79	86	73	75	93	92	92
82	88	73	75	94	92	93

TABLE XXV

Reflectance and 60° Gloss of Formula No. AF-67 after 100 hours at 500°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
2	90	76	78	92	91	89
5	90	77	79	93	90	90
8	90	78	79	88	88	87
11	90	77	78	91	90	89
26	89	76	78	85	87	85
29	90	78	79	86	90	90
32	90	79	79	86	90	89
35	91	77	79	86	89	89
50	90	77	79	92	90	89
53	90	78	79	91	89	87
56	90	78	79	91	90	89
59	90	78	79	91	90	89
74	90	77	78	94	90	90
77	90	77	78	91	90	89
80	90	78	80	93	91	90
83	91	80	81	86	82	82

TABLE XXVI

Reflectance and 60° Gloss of Formula No. AF-68-5 after 100 hours at 500°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
3	91	82	82	85	80	77
6	92	81	83	85	79	77
9	91	82	84	85	79	78
12	91	82	84	85	79	78
27	92	81	82	92	79	77
30	92	82	83	91	80	78
33	91	83	84	93	77	76
36	92	83	84	86	79	78
51	91	82	83	85	79	76
54	92	82	83	35	80	78
57	92	83	84	85	79	78
60	91	82	84	86	79	77
75	90	82	82	89	79	78
78	91	82	84	86	80	78
81	91	82	84	84	80	77
84	91	82	83	85	77	76

TABLE XXVII

Reflectance and 60°Gloss of Formula No AF-47 after 100 hours at 550°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60°Gloss Orig.	60°Gloss 50 hrs.	60°Gloss 100 hrs.
13	80	81	*	88	85	*
16	80	*	--	87	*	--
19	81	*	--	87	*	--
22	80	*	--	87	*	--
37	79	81	*	87	86	*
40	80	81	*	88	87	*
43	80	*	--	86	*	--
46	80	*	--	85	*	--
61	80	*	--	86	*	--
64	79	*	--	86	*	--
67	80	*	--	86	*	--
70	81	*	--	86	*	--
85	79	81	*	88	85	*
88	80	*	--	86	*	--
91	81	*	--	88	*	--
94	80	*	--	88	*	--

* FAILURE

TABLE XXVIII

Reflectance and 60°Gloss of Formula No. AF-58 after 100 hours at 550°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60° Gloss Orig.	60° Gloss 50 hrs.	60° Gloss 100 hrs.
14	82	83	84	82	79	80
17	83	84	84	80	78	78
20	82	84	85	81	79	79
23	82	84	84	81	80	77
38	82	84	85	81	80	80
41	83	84	85	80	79	76
44	83	84	85	81	80	79
47	83	84	84	80	80	73
62	82	83	84	82	80	79
65	83	84	84	81	80	79
68	83	85	85	81	79	80
71	83	84	84	80	79	78
86	82	83	84	81	78	78
89	82	84	85	82	80	79
92	83	84	85	81	79	78
95	83	84	84	82	80	79

TABLE XXIX

Reflectance and 60°Gloss of Formula No. AF-58-5 after 100 hours at 550°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs	60°Gloss Orig.	60°Gloss 50 hrs.	60°Gloss 100 hrs.
15	85	86	*	82	79	*
18	84	*	--	82	*	--
21	85	*	--	80	*	--
24	86	*	--	82	*	--
39	85	86	*	82	81	*
42	85	*	--	81	*	--
45	86	*	--	82	*	--
48	85	*	--	80	*	--
63	85	86	*	80	78	*
66	85	*	--	82	*	--
69	86	*	--	82	*	--
72	86	*	--	82	*	--
87	85	86	*	81	78	*
90	85	*	--	80	*	--
93	85	*	--	80	*	--
96	85	*	--	83	*	--

* FAILURE

TABLE XXX

Reflectance and 60°Gloss of Formula No. AF-66 After 100 hours at 550°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60°Gloss Orig.	60°Gloss 50 hrs.	60°Gloss 100 hrs.
1	75	77	78	90	90	90
4	75	77	78	90	90	90
7	76	78	79	90	88	89
10	75	78	78	90	89	89
25	75	77	78	90	90	89
28	76	77	78	90	89	89
31	76	78	79	90	88	88
34	76	78	79	90	88	88
49	75	77	78	88	87	87
52	76	78.	78	89	89	88
55	77	78	79	89	87	87
58	77	78	79	90	89	88
73	75	77	78	91	89	89
76	74	77	78	91	90	90
79	75	77	78	92	90	90
82	75	78	78	93	91	91

TABLE XXXI

Reflectance and 60°Gloss of Formula No. AF-67 after 100 hours at 550°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
2	78	80	80	89	86	86
5	79	80	80	90	87	87
8	79	80	81	87	85	84
11	78	80	80	89	86	86
26	78	80	80	85	83	83
29	79	80	81	90	88	87
32	79	81	82	89	87	87
35	79	80	81	89	88	87
50	79	80	80	89	86	84
53	79	80	81	87	86	85
56	79	80	81	89	87	84
59	79	80	81	89	88	87
74	78	80	80	90	87	85
77	78	80	80	89	86	85
80	80	81	81	90	88	87
83	81	83	84	82	80	80

TABLE XXXII

Reflectance and 60°Gloss of Formula No. AF-68-5 after 100 hours at 550°F

Panel No.	Reflectance			60°Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
3	82	84	84	77	72	72
6	83	84	84	77	73	72
9	84	84	85	78	74	73
12	84	84	84	78	74	74
27	82	83	84	77	74	72
30	83	84	85	78	75	75
33	84	85	86	76	72	71
36	84	84	85	78	74	73
51	83	84	84	76	73	72
54	83	84	85	78	74	73
57	84	84	85	78	74	73
60	84	84	85	77	74	72
[illegible]	82	84	84	78	72	72
78	84	84	85	78	76	73
81	84	85	85	77	74	72
84	83	84	84	76	71	70

TABLE XXXIII

Reflectance and 60°Gloss of Formula No. AF-58 after 100 hours at 600°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60° Gloss Orig.	60° Gloss 50 hrs.	60° Gloss 100 hrs.
14	84	84	*	80	78	*
17	84	*	--	78	*	--
20	85	*	--	79	*	--
23	84	*	--	77	*	--
38	85	*	--	80	*	--
41	85	*	--	76	*	--
44	85	*	--	79	*	--
47	84	*	--	73	*	--
62	84	85	*	79	78	*
65	84	*	--	79	*	--
68	85	85	*	80	78	*
71	84	*	--	78	*	--
86	84	84	*	78	77	*
89	85	85	*	79	78	*
92	85	85	*	78	76	*
95	84	85	*	79	78	*

* FAILURE

TABLE XXXIV

Reflectance and 60°Gloss of Formula No. AF-66 after 100 hours at 600°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60° Gloss Orig.	60° Gloss 50 hrs.	60° Gloss 100 hrs.
1	78	80	80	90	89	87
4	78	80	80	90	88	87
7	79	80	81	89	87	86
10	78	80	80	89	87	86
25	78	79	80	89	87	86
28	78	79	80	89	87	86
31	79	80	80	88	87	85
34	79	80	80	88	87	85
49	78	78	79	87	84	84
52	78	79	80	88	83	85
55	79	80	80	87	86	84
58	79	79	80	88	87	87
73	78	79	79	89	88	86
76	78	80	79	90	89	88
79	78	79	79	90	83	86
82	78	80	79	91	90	89

TABLE XXXV

Reflectance and 60° Gloss of Formula No. AF-67 after 100 hours at 600°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
2	80	81	82	86	83	81
5	80	82	82	87	85	82
8	81	82	82	84	81	79
11	80	82	82	86	83	82
26	80	80	82	83	79	78
29	81	81	82	87	85	83
32	82	82	83	87	85	83
35	81	81	82	87	85	83
50	80	80	81	85	82	82
53	81	81	82	86	82	83
56	81	81	82	85	83	81
59	81	81	81	87	85	84
74	80	81	81	86	84	84
77	80	81	81	86	85	83
80	81	82	81	87	85	84
83	84	85	*	80	79	*
* FAILURE						

TABLE XXXVI

Reflectance and 60° Gloss of Formula No. AF-68-5 after 100 hours at 600°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
3	84	86	85	72	69	68
6	84	85	85	72	69	67
9	85	86	85	73	71	69
12	84	86	85	74	71	69
27	84	84	85	72	69	68
30	85	85	85	75	72	70
33	86	85	86	71	67	66
36	85	85	86	73	69	69
51	84	84	84	72	68	68
54	85	84	85	73	69	69
57	85	85	85	73	70	70
60	85	85	85	72	71	70
75	84	84	84	72	68	68
78	85	85	84	73	71	70
81	85	85	85	72	70	69
84	84	85	84	70	68	66

TABLE XXXVII

Reflectance and 60°Gloss of Formula No. AF-66 after 100 hours at 650°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60° Gloss Orig.	60° Gloss 50 hrs.	60° Gloss 100 hrs.
1	80	82	82	87	85	83
4	80	82	82	87	85	84
7	81	82	83	86	84	80
10	80	82	82	86	85	82
25	80	81	82	86	82	78
28	80	81	82	86	85	82
31	80	82	83	85	84	81
34	80	82	82	85	83	80
49	79	81	82	84	82	80
52	80	81	83	85	80	77
55	80	82	83	84	81	77
58	80	81	82	87	83	80
73	79	81	84	86	84	80
76	79	81	83	88	85	81
79	79	81	82	86	84	81
82	79	81	82	89	87	84

TABLE XXXVIII

Reflectance and 60°Gloss of Formula No. AF-67 after 100 hours at 650°F

Panel No.	Reflectance Orig.	Reflectance 50 hrs.	Reflectance 100 hrs.	60° Gloss Orig.	60° Gloss 50 hrs.	60° Gloss 100 hrs.
2	82	83	83	81	79	77
5	82	83	84	83	80	77
8	82	83	83	79	76	72
11	82	83	83	82	79	76
26	82	82	83	78	76	73
29	82	83	84	83	79	76
32	83	83	84	83	80	77
35	82	83	84	83	80	76
50	81	82	83	82	79	74
53	82	83	83	83	79	76
56	82	83	83	81	79	76
59	81	82	83	84	81	77
74	81	83	83	84	79	76
77	81	83	83	83	80	77
80	81	83	83	84	80	75

TABLE XXXIX

Reflectance and 60° Gloss of Formula No. AF-68-5 after 100 hours at 650°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
3	85	86	*	68	64	*
6	85	*	--	67	*	--
9	85	*	--	69	*	--
12	85	*	--	69	*	--
27	85	*	--	68	*	--
30	85	*	--	70	*	--
33	86	*	--	66	*	--
36	86	*	--	69	*	--
51	84	86	*	68	64	*
54	85	*	--	69	*	--
57	85	*	--	70	*	--
60	85	*	--	70	*	--
75	84	86	*	68	65	*
78	84	86	*	70	66	*
81	85	86	*	69	65	*
84	84	*	--	66	*	--

* FAILURE

TABLE XL

Reflectance and 60° Gloss of Formula No. AF-66 after 100 hours at 700°F

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
1	82	*	--	83	*	--
4	82	*	--	84	*	--
7	83	*	--	80	*	--
10	82	*	--	82	*	--
25	82	83	*	78	75	*
28	82	83	*	82	79	*
31	83	84	*	81	80	*
34	82	*	--	80	*	--
49	82	83	*	80	80	*
52	83	*	--	77	*	--
55	83	*	--	77	*	--
58	82	*	--	80	*	--
73	84	85	*	80	77	*
76	83	*	--	81	*	--
79	82	*	--	81	*	--
82	82	*	--	84	*	--

*FAILURE

TABLE XLI

Reflectance and 60° Gloss of Formula No. AF-67 after 100 hours at $700^\circ F$

Panel No.	Reflectance			60° Gloss		
	Orig.	50 hrs.	100 hrs.	Orig.	50 hrs.	100 hrs.
2	83	*	--	77	*	--
5	84	*	--	77	*	--
8	83	*	--	72	*	--
11	83	*	--	76	*	--
26	83	*	--	73	*	--
29	84	*	--	76	*	--
32	84	*	--	77	*	--
35	84	*	--	76	*	--
50	83	*	--	74	*	--
53	83	*	--	76	*	--
56	83	*	--	76	*	--
59	83	*	--	77	*	--
74	83	*	--	76	*	--
77	83	*	--	77	*	--
80	83	*	--	75	*	--

* FAILURES

TABLE XLII

CODE SHEET FOR TYPE OF COATING FAILURE AFTER HIGH TEMPERATURE EXPOSURE

Panel No.	Failure	Panel No.	Failure	Panel No.	Failure
AF-1	Mod F	AF-33	Sv C	AF-65	Mod F
AF-2	Sv F	AF-34	Sv F C	AF-66	V S C
AF-3	Mod F	AF-35	Sv F	AF-67	V S P
AF-4	Sv F	AF-36	Sv C Mod P	AF-68	C P
AF-5	Sv F	AF-37	Sm CF	AF-69	S C
AF-6	Sv P	AF-38	Sm CF	AF-70	S P
AF-7	S P	AF-39	Lg CF	AF-71	S F
AF-8	Sv F	AF-40	Lg A	AF-72	V S C
AF-9	Sv P	AF-41	Mod. CF	AF-73	S P
AF-10	Sv F	AF-42	S C	AF-74	V S P C
AF-11	Sv F	AF-43	S C	AF-75	Sv P
AF-12	Sv P	AF-44	Mod. A Mod F	AF-76	Sv P
AF-13	Mod CF	AF-45	Mod A	AF-77	Sv F
AF-14	C	AF-46	Mod P	AF-78	Sv P
AF-15	Sm CF	AF-47	SP	AF-79	Sv C
AF-16	S F	AF-48	A	AF-80	Sv P
AF-17	Mod P	AF-49	S P	AF-81	Sv P
AF-18	C & P	AF-50	Mod C	AF-82	Sv CF
AF-19	S P	AF-51	Sv A	AF-83	S C
AF-20	Mod P	AF-52	P	AF-84	C P
AF-21	V S C	AF-53	Sv F	AF-85	Lg A
AF-22	V L C	AF-54	Sv P	AF-86	V V S C
AF-23	Sv P	AF-55	Sv F	AF-87	Mod CF
AF-24	C F S P	AF-56	Sv P	AF-88	SP
AF-25	S P	AF-57	Sv P	AF-89	Spot C
AF-26	Mod C	AF-58	Sv C	AF-90	V S C
AF-27	Mod A	AF-59	Sv F	AF-91	S P
AF-28	S P	AF-60	C P	AF-92	Mod P
AF-29	Sv F	AF-61	V S C	AF-93	V S C
AF-30	Sv P	AF-62	Sv A	AF-94	Mod P
AF-31	S P	AF-63	Lg CF	AF-95	S C P
AF-32	SvF	AF-64	V S C	AF-96	V S C

CODE

A	Alligatoring	P	Peeling
C	Cracking	S	Slight
CF	Crow-footing	Sm	Small
F	Flaking	Sv	Severe
L	Light	V	Very
Lg.	Large		
M	Moderate		

APPENDIX IV

High Temperature Evaluation After 1000 Hours at 460° F

APPENDIX IV

High Temperature Evaluation After 1000 Hours at 460°F

TABLE XLIII

60° Gloss of Formula No. AF-47

Panel No.	HOURS 0	250	500	750	1000
13	88	78	78	78	80
37	90	82	82	81	82
61	85	81	80	80	81
85	93	80	81	80	81

TABLE XLIV

60° Gloss of Formula No. AF-58

Panel No.	HOURS 0	250	500	750	1000
14	83	76	74	75	75
38	83	75	74	75	73
62	83	73	75	75	76
86	88	74	74	76	75

TABLE XLV

60° Gloss of Formula No. AF-58-5

Panel No.	HOURS 0	250	500	750	1000
15	87	77	74	75	76
39	86	75	74	75	73
63	85	74	73	74	75
87	90	75	76	77	76

TABLE XLVI

60° Gloss of Formula No. AF-66

Panel No.	HOURS 0	250	500	750	1000
1	89	82	83	85	85
25	88	81	82	83	83
49	91	82	82	81	82
73	97	83	80	80	81

TABLE XLVII

60°Gloss of Formula No. AF-67

Panel No.	HOURS 0	250	500	750	1000
2	92	80	80	82	83
26	90	80	80	82	83
50	90	80	80	80	81
74	94	84	82	81	82

TABLE XLVIII

60°Gloss of Formula No. AF-68-5

Panel No.	HOURS 0	250	500	750	1000
3	85	69	70	70	70
27	84	72	71	71	71
51	82	66	67	66	65
75	91	69	69	69	69

TABLE XLIX

Reflectance of Formula No. AF-47

Panel No.	HOURS 0	250	500	750	1000
13	86	82	81	81	82
37	87	82	82	81	82
61	87	82	82	81	83
85	86	81	81	82	81

TABLE L

Reflectance of Formula No. AF-58

Panel No.	HOURS 0	250	500	750	1000
14	90	85	84	85	84
38	91	85	84	83	85
62	89	84	84	83	85
86	90	84	85	85	84

TABLE LI

Reflectance of Formula No. AF-58-5

Panel No.	HOURS 0	250	500	750	1000
15	90	87	85	86	86
39	87	86	84	85	85
63	89	86	85	84	85
87	89	86	86	86	85

TABLE LII
Reflectance of Formula No. AF-66

Panel No.	HOURS 0	250	500	750	1000
1	87	78	78	80	80
25	87	79	78	79	80
49	86	79	78	78	79
73	86	79	79	80	80

TABLE LIII
Reflectance of Formula No. AF-67

Panel No.	HOURS 0	250	500	750	1000
2	90	82	81	82	82
26	90	82	81	82	83
50	90	82	82	81	82
74	89	81	82	83	82

TABLE LIV
Reflectance of Formula No. AF-68-5

Panel No.	HOURS 0	250	500	750	1000
3	91	86	84	84	84
27	91	86	85	84	85
51	91	85	85	84	85
75	89	85	85	86	85

APPENDIX V

Fuel Resistance

APPENDIX V
Fuel Resistance

Table LV

Results of Fuel Resistance Evaluation

Formula	MIL-L-7808*		Type II Fluid**		TT-T-656***	
	Before	After	Before	After	Before	After
AF-66	HB	6B	HB	4B	HB	6B
AF-67	HB	6B	HB	4B	HB	6B
AF-68-5	HB	6B	HB	4B	HB	6B
AF-47	HB	B-2B	HB	4B	HB	B-2B
AF-58	HB	B-2B	HB	4B	HB	B-2B
AF-58-5	HB	B-2B	HB	4B	HB	B-2B

* Specification MIL-L-7808 Synthetic Lubricant.
** Hydrocarbon Test Fluid, Type II.
*** Specification TT-T-636 Tricresyl Phosphate.

Table LVI

Pencil Hardness after Immersion and Heat Cure of Formulations No. AF-47 and AF-66

Panel No.	Formula	Test Fluid	Original	After	30 Min. at 460°F
1	AF-66	MIL-L-7808	HB	6B	HB
1	AF-66	Type II	HB	4B	HB
1	AF-66	TT-T-656	HB	6B	HB
13	AF-47	MIL-L-7808	HB	B-2B	HB
13	AF-47	Type II	HB	4B	HB
13	AF-47	TT-T-656	HB	B-2B	HB

APPENDIX VI

Weatherometer Exposure

APPENDIX VI
Weatherometer Exposure

TABLE LVII

Reflectance and 60°Gloss of Formula No. AF-47 after 500 hours Exposure in the Weather-ometer.

Panel No.	Reflectance Orig.	Reflectance 250	Reflectance 500	60°Gloss Orig.	60°Gloss 250	60°Gloss 500
13	86	87	86	89	86	87
16	87	87	86	87	83	84
19	87	86	86	88	85	86
22	86	87	86	89	84	85
37	86	87	86	89	86	86
40	86	87	86	91	86	86
43	86	85	85	89	85	85
46	88	87	87	90	88	88
61	86	84	85	88	90	86
64	88	86	87	87	90	86
67	86	85	85	88	90	87
70	87	86	86	88	88	87
85	87	86	87	89	89	87
88	87	86	86	86	89	86
91	88	87	87	90	90	87
94	88	87	87	90	88	88

TABLE LVIII

Reflectance and 60°Gloss of Formula No. AF-58 after 500 hours Exposure in the Weather-ometer.

Panel No.	Reflectance Orig.	Reflectance 250	Reflectance 500	60°Gloss Orig	60°Gloss 250	60°Gloss 500
14	90	90	90	85	80	81
17	90	90	90	82	78	79
20	90	90	90	82	79	80
23	90	90	89	83	79	80
38	91	90	89	84	82	81
41	90	89	89	83	81	81
44	91	89	89	85	82	82
47	91	90	90	84	82	83
62	91	89	90	85	85	80
65	90	91	91	85	84	81
68	90	91	91	85	85	83
71	89	91	90	85	84	80
86	90	90	90	85	85	83
89	89	90	90	83	85	82
92	90	90	90	84	84	81
95	91	90	90	87	85	83

TABLE LIX

Reflectance and 60°Gloss of Formula No. AF-58-5 after 500 hours Exposure in the Weather-ometer.

Panel No.	Reflectance			60°Gloss		
	Orig.	250	500	Orig.	250	500
15	90	90	90	87	82	82
18	90	90	90	87	82	82
21	89	90	90	86	82	82
24	87	90	90	88	84	85
39	91	90	90	87	85	85
42	90	90	89	87	84	83
45	91	90	90	89	85	85
48	91	90	90	87	86	86
63	91	89	90	87	88	84
66	91	90	90	88	86	83
69	92	90	90	88	86	85
72	91	90	90	87	87	85
87	90	89	90	87	84	84
90	90	89	89	87	85	83
93	90	89	89	87	86	84
96	91	89	90	89	85	83

TABLE LX

Reflectance and 60°Gloss of Formula No. AF-66 after 500 hours Exposure in the Weather-ometer.

Panel No.	Reflectance			60°Gloss		
	Orig.	250	500	Orig.	250	500
1	88	86	86	91	88	87
4	89	86	86	93	88	88
7	87	87	87	91	91	91
10	88	87	87	92	91	90
25	88	87	86	90	78	83
28	87	88	87	93	80	86
31	87	87	86	93	87	89
34	89	87	86	91	88	90
49	88	86	87	88	90	88
52	88	87	88	92	91	88
55	88	86	87	89	91	89
58	88	86	87	92	90	87
73	86	86	87	92	92	90
76	87	86	86	92	91	89
79	86	85	85	93	91	91
82	88	86	87	94	94	92

TABLE LXI

Reflectance and 60°Gloss of Formula No. AF-67 after 500 hours Exposure in the Weather-ometer

Panel No.	Reflectance Orig.	Reflectance 250	Reflectance 500	60°Gloss Orig.	60°Gloss 250	60°Gloss 500
2	90	89	90	92	85	87
5	90	89	90	93	88	88
8	90	89	89	88	88	90
11	90	89	89	91	90	90
26	89	90	89	85	80	86
29	90	90	89	86	80	84
32	90	90	89	86	84	87
35	91	90	89	86	86	88
50	90	88	89	92	89	86
53	90	89	90	91	90	86
56	90	89	90	91	91	89
59	90	89	90	91	90	87
74	90	89	90	94	91	88
77	90	89	89	91	90	88
80	90	89	89	93	93	90
83	91	91	91	86	87	84

TABLE LXII

Reflectance and 60°Gloss of Formula No. AF-68-5 after 500 hours Exposure in the Weather-ometer

Panel No.	Reflectance Orig.	Reflectance 250	Reflectance 500	60°Gloss Orig.	60°Gloss 250	60°Gloss 500
3	91	90	91	85	81	82
6	92	91	91	85	81	82
9	91	90	91	85	83	82
12	91	90	91	85	82	82
27	92	90	90	92	78	80
30	92	91	89	91	79	80
33	91	90	90	93	81	80
36	92	91	90	86	86	81
51	91	91	91	85	82	81
54	92	89	90	85	85	82
57	92	90	90	85	84	81
60	91	90	90	86	85	82
75	90	89	90	89	88	85
78	91	91	91	86	85	82
81	91	90	91	84	85	82
84	91	90	90	85	85	82

APPENDIX VII

Raw Materials Data

TABLE LXIII

Silicone Resin DC-808*

Properties	
Solids Content %	50%
Solvent	Xylene
Thin with	Xylene
Specific Gravity (25°C, 77°F)	1.01
Weight per Gallon (lbs)	8.4
Viscosity (centipoises at 25°C)	100 - 200
Gardner-Holdt	D-H
Color	Straw
Gardner	2

*Dow Corning Corporation, Midland, Michigan, 48640

TABLE LXIV

Silicone Resin SR-125*

Properties	
Solids Content %	50 ± 1
Solvent	Xylol
Thin with	Xylol or other aromatic hydrocarbons
Specific Gravity (25°C, 77°F)	1. (avg.)
Weight per Gallon (lbs.)	8.34 (avg.)
Viscosity (centipoises at 25°C)	80-150
Gardner-Holdt	C-F
Catalyst Content	None
Color	Light straw
Gardner	0-1
Storage Stability (at 100°F)	6 months
Flash Point	
(Tag (Open Cup)	Above 80°F
Suggested Curing Time	1 hour at 480°F

*Mfg by General Electric, Silicone Products Department, Waterford, New York.

TABLE LXV

Silicone Resin SR-223*

Properties	
Solids Content %	60 ± 1
Solvent	Toluene
Thin with	Xylol or other aromatic hydrocarbons
Specific Gravity (25°C, 77°F)	1.04 (avg.)
Weight per Gallon (lbs)	8.7
Viscosity (centipoises at 25°C) Brookfield Model RFV**	175-275
Gardner-Holdt	E-H
Color	Straw
Gardner	6
Storage Stability (at $<$ 100°F)	6 months
Flash Point (closed cup)	
ASTM D56-36	35°F

*Mfg by General Electric, Silicone Products Department, Waterford, New York.

**Using a #1 spindle at 20 rpm

TABLE LXVI

Reduced Zinc Molybdate*
No. 0831

Percent Zn_2MoO_4	18-22
Balance	$CaCO_3$
Percent Moisture (at 110°C)	Max. 0.3
Percent Water Soluble $SO_4^=$	Max. 0.1
Oil Absorption	25
Specific Gravity	3.00
Weight per solid gallon (lb.)	24.99
One pound bulks (gal.)	.04002

*Manufactured by Mineral Pigments Corporation, Muirkirk, Maryland.

TABLE LXVII

Properties of Z-6020*

Z-6020, N(trimethoxysilylpropyl) ethylenediamine, is an amino-functional material with the following properties.

Typical Physical Properties

Physical Form	Amber
Boiling Point (15 mm Hg)	146°C
Specific Gravity 25°C/25°C	1.045
Flash Point	250°F
Solvents	Benzene, ethyl ether, methyl alcohol

* Dow Corning Corporation, Chemical Products Division, Midland, Michigan, 48640

TABLE LXVIII

Properties of A-1100 Silane*

A-1100, gama-aminopropyltriethoxysilane, is an amino-functional material with the following properties:

Typical Physical Properties

Physical Form	Water white liquid
Boiling Point (30 mm. Hg)	123°C
(760 mm. Hg)	217°C
Specific Gravity, 25°C/25°C	0.94
Flash Point (COC)	200°F
Solvents	Benzene, methyl cellosolve, chloroform dioxane, ethanol, heptane, toluene, water (by hydrolysis)

*Union Carbide Corporation, Silicones Division, 270 Park Avenue, New York, New York 10017

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13. ABSTRACT

Improved high temperature protective coatings primarily for use on high speed Mach 3 aircraft and missiles have been developed which are capable of withstanding the extreme environments and aerodynamic heating. By selectively incorporating aminosilances as catalysts for curing unmodified polymethylphenyl silicone resins, air dry (ambient temperature), stable coatings with retained reflectances exceeding eighty (80) percent after elevated temperature exposures were developed. Analysis of two years Florida weathering data indicates that these coatings when properly applied to titanium, stainless steel and aluminum alloys have excellent adhesion, corrosion resistance, and are extremely resistant to solar discoloration thus making them excellent candidates for high speed aircraft and missiles. A variety of air dry silicone primer systems were also developed, evaluated for thermal stability and corrosion resistance, and optimized for the best topcoats formulated. Based on the laboratory and Florida weathering results, a silicone-base coating system which will dry under ambient temperature conditions (75 ± 2°F), and servicable for use up to 700°F for short periods and 600°F for prolonged periods has been developed

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Air Dry Coatings						
White Pigmented						
700° F						
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